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## SOME EFFECTS OF VARYING SUGAR CONCENTRATIONS IN BREAD DOUGH ON FERMENTATION BY-PRODUCTS AND FERMENTATION TOLERANCE <sup>1</sup>

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Many investigators, among the first of whom is Wood (1907), have recognized that in the making of yeast-leavened bread there are two distinct factors that predominate in determining the character of the finished loaf. One is gas production and the other is gas retention. The gas-producing agents in bread dough are, of course, yeast and sugar. The three sources of sugar in the dough are, first, sugar originally present in the flour; second, the flour diastases which liquefy starch and gradually convert it into maltose; and third, the amount of sugar intentionally added as a dough ingredient. The manner in which the dough retains the gas that is produced by yeast fermentation depends upon both the quantity and the structural properties or so-called "quality" of the gluten.

Efficient utilization of these two factors in bread production is dependent upon the manipulation of certain environmental factors, chief among which are temperature, time, ingredients, mechanical treatment, etc. Assuming a proper regulation of temperature and ingredients, the *fermentation time* is usually regarded as a most important and critical item.

Other things being equal, it is conceivable that varying the fermentation time alone will substantially affect both the gas production and the gas retention factors. As fermentation progresses a point of maximum rate of carbon dioxide production is reached, beyond which the rate of gas production progressively diminishes as the supply of available sugars becomes exhausted and diastatic activity falls off. The effect upon the gas-retaining agent, the gluten, whether of degradation or improvement, will presumably depend, at least in part, upon

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the influence of the following factors: Proteolytic enzymes, H-ion changes, development of acidity, and alcohol.

## Basis for Judgment of Fermentation Tolerance

An intelligent understanding of fermentation tolerance in bread production necessitates the establishment of a sound basis for recognizing the extent to which both the gas production and the gas retention factors, individually, and in relation to each other, are affected by fermentation time alone.

The standard A. A. C. C. baking test procedure offers highly satisfactory means for distinguishing and measuring both factors, despite the type of criticism and objection recently advanced by Jørgensen (1931). He, and others cited by him, point out that the A.A.C.C. basic procedure is a rather severe tax on the diastatic or "gassing" power of the flour, and that in cases where this happens to be deficient, a shortage of fermentable sugars in the later stages of fermentation will inevitably result in an inferior test loaf, thereby obscuring other good qualities that the flour may possess. The gluten quality or "strength" of the flour should always have an opportunity to manifest itself, and, therefore, the test procedure should be such as to always insure an adequate maintenance of gas production throughout the entire fermentation period. As a safeguard against the likelihood of a shortage of fermentable sugar, Jørgensen proposes a reduction of the yeast to 1% instead of 3% as now specified.

It is not our present purpose to justify the use of 3% instead of 1% of yeast, although there are good reasons in favor of 3%. What Jørgensen and other critics of the standard A. A. C. C. procedure apparently fail to appreciate is that the so-called "basic procedure method" is merely a reference method, and is, as pointed out by Werner (1925), a "foundation for subsequent tests." The method as adopted by the A. A. C. C. distinctly embodies "Additional and Supplementary" tests. Four such tests are indicated, but it should require no great stretch of the imagination to recognize that other supplementary tests might well have been included. The suggestion of supplementary tests is merely in recognition of the obvious fact that starting from an established foundation it is permissible to vary any one factor at a time without violating any principles of scientific laboratory testing.

As a matter of fact, there are probably few laboratories which do not use one of the so-called supplementary methods in daily routine work. For flours of low diastatic power there is a choice of several procedures whereby Jørgensen's (1931) objections may be easily overcome. Reduction of yeast percentage, reduction of time (see

supplementary procedure B), reduction of temperature, or an increased amount of sugar added to the dough; any one of these four will accomplish the desired result. Blish and Sandstedt (1927) used increased amounts of sugar for low diastatic flours. Markley and Bailey (1931) prefer to add small quantities of sprouted wheat flour. Blish, Sandstedt, and Platenius (1929) have shown that in any specified, arbitrarily fixed, and properly controlled baking procedure there is a positive and dependable relationship between depth of crust color and diastatic or "gassing power." With this in mind, and assuming precision in the control of both degree and uniformity of fermentation and baking temperatures, it is possible even with the A. A. C. C. basic method alone to go a long way in interpreting the test loaf both from the standpoint of gluten properties and of "gassing power." A small, very pale loaf indicates exhaustion of the gas production factor, i.e., fermentable sugar, and necessitates re-baking by one of the supplementary procedures previously mentioned in order to permit gluten properties to register. If the loaf has good crust color but is inferior in other respects, then the inferiority is due to the "strength" (quality of condition of the gluten), and not to lack of "gassing power." That crust color alone as a diagnostic symptom has not received the attention that its importance justifies is probably due to a lack of the necessary precision in oven temperature control and uniformity on the part of the majority of technicians.

All things considered, there is justification for the belief that the criticism of Jørgensen (1931) and of others is based upon an inadequate understanding and appreciation of the intent, purpose and scope of the present standard A. A. C. C. experimental baking test. There is no intention of restricting the operator to the basic procedure alone; supplementary procedures are contemplated and are to be encouraged. To the intelligent operator, however, the basic procedure, when properly employed, will frequently give all necessary information. At its worst, it will in the great majority of instances indicate just which of the many possible supplementary procedures should be used in the securing of additional necessary information.

In the standard baking test the characteristic symptoms of deficient gas production are a limited rise in the pan during proof, failure of the dough to rise or "spring" in the oven, and an exceedingly pale crust color in the baked loaf. There is also a frequent tendency toward a slight raggedness or pulling away of the top crust. The character of the gluten, the gas-retaining structure of the dough, is also judged from certain loaf characteristics, among which are size and shape, raggedness or smoothness of the loaf exterior, and firmness, grain, texture, and coherence of the crumb.

Since the pioneer work of Wood (1907), many workers have recognized the importance of distinguishing between the factors of gas production and gas retention (gluten properties), both in commercial bread production and in laboratory flour testing. The separation of the two factors from each other is regarded by Fisher and Halton (1929) as "perhaps the biggest positive advance that has yet been made in the study of strength."

While duly recognizing the importance of distinguishing between these two factors, the question is sometimes raised as to the *extent* to which they are individually and respectively associated with fermentation tolerance. There is a prevailing impression,—to a greater degree among practical commercial bakers and to some extent among laboratory technicians,—that when fermentation time is prolonged beyond a so-called "optimum" there is a gradual and progressive degrading influence upon both "gassing power" and gluten strength, with emphasis usually on the latter. To what extent is this true, and to what degree, if any, are the two factors mutually dependent upon each other?

Experiences in this laboratory thus far indicate that when baking with simple ingredients the two factors are independent. Blish and Sandstedt (1927), using the standard A. A. C. C. baking test procedure, found no evidence of any serious gluten degradation with increasing fermentation time. It was true, of course, that the gluten characteristics largely governed the type of loaf produced, but the time range over which the doughs would tolerate fermentation without the appearance of the usual "symptoms of age" in the bread was found to depend chiefly upon the maintenance of gas production (fermentable sugars) in the dough. This time range could be reduced to a minimum by excluding sugar from the dough ingredients, in which event the sole source of sugar is the natural diastatic power of the flour. On the other hand, it was invariably found possible to prolong the fermentation time, with corresponding postponement of the usual "symptoms of age" to a degree directly proportional to the quantity of sugar added in the dough ingredients.

These findings led to the conclusions that fermentation tolerance is almost exclusively a function of the gas-producing agencies in the dough; that in an arbitrarily fixed and controlled straight dough baking procedure (such as the standard A. A. C. C. procedure) variations in fermentation tolerance merely reflect corresponding variations in diastatic power, that in this type of baking crust color becomes an index to diastatic power, that gluten is far more resistant to ordinary yeast fermentation than it is generally given credit for, and that this general situation explains why the sponge dough method of bread

making (in which the sugar is incorporated almost immediately preceding panary fermentation, thereby insuring a maintained gas production) is more fool proof than the straight dough method. These laboratory findings and conclusions were supported and verified by experiments conducted under commercial shop conditions.

It is to be noted that this situation and these conclusions apply only to *sound* flour baked with the simple ingredients—yeast, sugar and salt. In cases of unsound flour, and more especially when diastatic malt or sprouted wheat preparations are used, prolonged fermentation may be expected to produce progressive degradation of the gluten. Possibly a considerable portion of the prevailing idea that the fermentation tolerance of *gluten* is limited, is based upon experience involving the use of diastatic malt, which was formerly a more common and general practice than at present, and which contains proteolytic enzymes. There is also, of course, the possibility that wheats of certain types and from certain localities may have abnormally high proteolytic activity. However, no such wheats have ever been encountered among the many samples of hard winter and spring varieties or types studied in this laboratory over a period of several years.

Object of the Investigation

As previously mentioned, the view that the gas production and gas retention factors are mutually independent, and that fermentation tolerance is entirely a function of the former, is not shared by all cereal technologists. That H-ion concentration is an important factor is proposed by a considerable group, including Jessen-Hansen (1911), Cohn, Cathcart, and Henderson (1918), Sorenson (1924), Dunlap (1926), and others. Other workers, including Grewe and Bailey (1927), and Fisher and Halton (1929), minimize the importance of H-ion concentration. Working (1929) lists the following factors as "probably being among the most important affecting dough fermentation or dough condition: Mechanical action; water; salts; acids; alcohol, fusel-oil, and other end-products of fermentation; oxidizing agents; phosphatides; enzymes." Working (1929) also states his belief that "the greatest need of cereal chemistry is fundamental research into the changes taking place during dough fermentation."

That the gas production and gas retention factors may not be entirely independent of each other is suggested by Werner and Siedhoff (1929). They propose that when the supply of fermentable sugars in the fermenting dough becomes exhausted or deficient, alcoholic fermentation is stopped or retarded, and the products that exert a degrading influence on the gluten become more active and exert a greater influence than formerly. In other words, active alcoholic fermentation

has a protective influence on the gluten. If this is true, the factors of gas production and gas retention cannot be regarded as independent of each other.

The investigation here reported was undertaken primarily for the following reasons:

1. To secure detailed information—under conditions specified for the standard A. A. C. C. baking test, and over a long range of fermentation time—regarding the respective rates of development of some of the more conspicuous by-products of dough fermentation that are customarily considered to affect the structural properties of gluten; i.e., H-ion concentration, alcohol, total, volatile, and non-volatile acidity.

2. To study comparative rates of development of these fermentation by-products under conditions, respectively, of (a) "normal" sugar concentration, (b) sugar deficiency, and (c) sugar excess.

3. To correlate the findings, if possible, with actual baking observations, in an effort to establish (a) any important relationship that might exist between the factors of gas production and gas retention, (b) further evidence as to the comparative importance of these factors in their influence on fermentation tolerance and bread characteristics.

No effort was made to study proteolysis during fermentation, since it is now well established that this is not a factor of consequence in any ordinary methods of bread making, excepting where malt, sprouted wheat products, or unsound flour is involved. Convincing proof of this assertion is furnished by the work of Olsen and Bailey (1925), Harrel (1927), Blish and Sandstedt (1927), Cairns and Bailey (1928), Sharp and Gortner (1924), Baehr (1925), Sharp and Schreiner (1926), and Brownlee and Bailey (1930).

## Experimental

Baking Procedure: A standard commercial "Bakers' Patent" flour was used throughout. Doughs were made according to the formula specified by Werner (1925) and were handled in general accordance with specifications of the standard A. A. C. C. basic procedure, with variations, only, as follows:

Doughs were made at three different levels of sugar concentration. In the first series no sugar was included among the dough ingredients; in the second series  $2\frac{1}{2}\%$  sugar (the amount specified in the basic procedure) was used, while in the third series 10% sugar was used. In each series bakings were made involving progressively increasing intervals of fermentation time, starting with no fermentation time and using 1 hour intervals of fermentation time thereafter. A constant proof period (55 minutes) was used in all bakes. In each series the

bakes were carried to the extreme limit of fermentation tolerance, as evidenced by failure of the dough to rise in the pan, loss of crust color with minimum loaf volume.

Quantitative estimations of the various fermentation by-products were made at regular intervals over a time range of 0 to 24 hours. All doughs with from 4 to 8 hours fermentation were "knocked down" at 2 hour intervals up to the time of the last "punch," which was 25 minutes before the molding or the withdrawing of samples for analysis. Doughs receiving more than 8 hours fermentation were mixed in the evening, covered and placed in the fermentation box, and left undisturbed until the following morning, when they were "knocked down" at regular 2 hour intervals. It was necessary to decrease the absorption about 3% in doughs standing overnight, and 5% in the ones with 10% initial sugar content.

Baking data and results are indicated in Table XIV, and in Figures 8, 9, and 10. In the figures illustrating the bread the numbers on the loaves represent hours of fermentation prior to the pan proof.

DETERMINATION OF ALCOHOL CONTENT OF DOUGH: A search through the literature revealed a lack of data on the progressive development of alcohol in the fermentation of bread dough. Simpson and Knight (1926) describe a method for the estimation of alcohol in baked bread, but this was found unsatisfactory for use with dough. After a number of procedures were tried and abandoned, the following method was adopted, whereby reliable and consistent results could be secured, even though a 100% recovery was not realized. The dough was broken up with a modified soda-fountain mixer described by Bouvoucous (1927) with 1600 cc. of water and 25 gms. of magnesium sulfate. This mixture was put into a round-bottomed flask, and distilled in an oil bath at 115-120° C. until 400 to 500 cc. of distillate came over, which usually required from 1½ to 2 hours. During the boiling in the flask, a current of air was passed through the mixture to prevent foaming and to aid in the removal of the alcohol. The distillate was collected in a liter flask immersed in a mixture of ice and salt. first distillate was made alkaline with CaO and redistilled to 250-300 cc. This distillate was again distilled into a 100 cc. volumetric flask, brought to a 100 cc. mark at 20° C., and specific gravity determined according to the A. O. A. C. method (1925).

Preliminary experiments were made to determine the amount of alcohol that could be recovered from known mixtures of alcohol and water; and also from mixtures of alcohol, water and fresh dough suspensions. These data are shown in Tables I and II.

Other methods that were attempted, but which proved unsatisfactory, included: (1) Steam distillation, (2) dry distillation with sand

in an oil bath at high temperatures, (3) ordinary distillation, (4) refluxing for an hour, followed by precipitation of the starch and distillation of the filtrate, and (5) distillation in an oil bath without the aid of air or magnesium sulfate.

TABLE I

AMOUNT OF ALCOHOL RECOVERED FROM ALCOHOL WATER SOLUTIONS

Alcohol added	Alcohol added	Alcohol recovered
cc.	P.ct. by vol.	P.ct. by vol.
25	23.40	23.38
25	23.55	23.50
10	9.55	9.53
5	4.80	4.73
5	4.70	4.68
2	1.90	1.89
2	1.93	1.90

TABLE II
PER CENT RECOVERY OF ALCOHOL ADDED TO DOUGHS

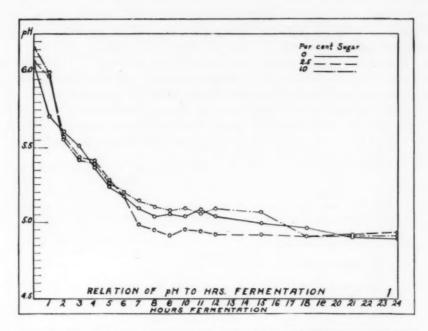
Per cent of total recovered	Alcohol recovered	Per cent of alcohol 1	Alcohol added
	P.ct.		cc.
92.65	22.32	23.45	25
89.11	8.43	9.46	10
93.26	4.43	4.75	5
94.76	1.81	1.91	2
	1.81	1.91	2
Average 92.45			

Based on weight of dough.

An additional procedure which gave satisfactory results was based upon a method used by Engle and Yoder (1926) to disperse soils for mechanical analysis. The dough was dispersed with the mechanical drink mixer and centrifuged. The clear liquid was poured into a distilling flask, the residue was thoroughly triturated with a glass rod, centrifuged, and the liquid added to the first. This procedure was repeated 3 times, and the clear liquid distilled as in the first procedure. Comparative results by the two satisfactory methods are shown in Table III.

Table VIII and Figure 2 record the progressive changes in alcohol concentration with fermentation time in the doughs of varying initial sugar content.

DETERMINATION OF HYDROGEN-ION CONCENTRATION: Progressive pH changes in the doughs of varying initial sugar concentration were studied, using a procedure as follows: 10 grams of dough were mixed with 100 cc. of water in the mechanical drink mixer, with a 3



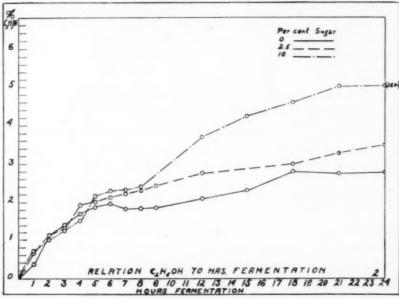


Fig. 1. Relation of pH to hours of fermentation.

Fig. 2. Relation of alcohol concentration to hours of fermentation.

minute stirring period. The suspension was then whirled in the centrifuge, and pH was determined in the clear liquid, using the Type K, Leeds and Northrup potentiometer; with a saturated calomel half cell and a Bailey (1920) type hydrogen electrode.

Preliminary experiments were carried out to show (1) the effect on pH of varying the ratio of dough to water, (2) the effect of centrifuging versus filtering through cotton, and (3) the effect on pH of punching the dough at 1 hour intervals as compared with no punching of the dough. The results of these experiments are shown in Tables IV, V and VI. The progressive changes in pH in the different doughs are set forth in Table VII, and in Figure 1.

TABLE III

Comparison of the Amount of Alcohol Recovered by Distilling Entire Dough Suspension and the Amount Recovered from the Centrifuged Liquid

Alcohol added	Dough suspension	Centrifuged liquid
Alcohol added	Alcohol recovered	Alcohol recovered
P.ct.	P.ct.	P.ct.
23.45	22.32	22.28
23.45	22.10	22.20
9.45	8.43	8.47
9.45	8.30	8.29
9.45 4.75	. 4.43	4.40
1.91	1.81	1.84
1.91	1.85	1.89

TABLE IV

RELATION OF THE AMOUNT OF DOUGH TO THE PH

Dough per 100 cc.	E.M.F.	pH
Gms.		-
5	0.615	6.25
8	.604	6.06
10	.601	6.01
15	.594	5.90
20	.610	6.17

TABLE V

COMPARISON OF CENTRIFUGE AND FILTER METHODS FOR PH

Time	Centrifuged	Filtered
Hrs.	pH	рН
1	6.17	6.17
3	5.44	5.46
3	5.20	5.17
10	4.98	4.93

TABLE VI Effect of Time of "Punch" on pH

Time	Punched	No punching
Hrs.	pH	pH
0	5.98	
1	5.98 5.69	5.69
4	5.37	5.69 5.35
6	5.30	5.29
8	4.97	4.95

TABLE VII

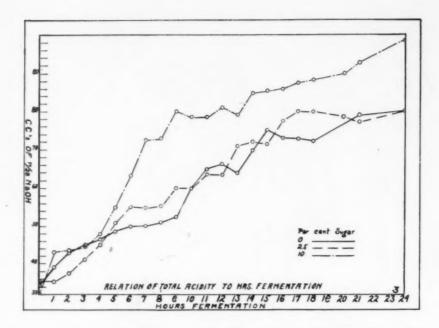
RELATION OF PH TO TIME OF FERMENTATION WITH VARYING SUGAR CONCENTRATIONS

Fermentation time	Per cent sugar		
	0	21	10
Hrs.	pH	pH	pł
0	6.03	6.07	6.1
1	5.74	5.97	5.9
2	5.61	5.57	5.5
2 3	5.51	5.44	5.4
4	5.37	5.41	5.3
4 5	5.24	5.28	5.2
6	5.17	5.17	5.2
6 7	5.09	4.98	5.1
8	5.03	4.95	5.1
8	5.05	4.92	5.0
10	5.03	4.95	5.0
11	5.08	4.94	5.0
12	5.03	4.92	5.0
15	4.98	4.92	5.0
18	4.95	4.91	4.9
21	4.90	4.92	4.9
24	4.88	4.93	4.9

DETERMINATION OF TOTAL ACIDITY OF DOUGH: Twenty grams of dough were disintegrated in 250 cc. of CO<sub>2</sub>-free distilled water, centrifuged, and 100 cc. portions were titrated with N/28 NaOH, using phenolphthalein as indicator.

The rate of development of acidity is shown in Table IX, and in Figure 3.

Determination of Total Acidity Minus CO<sub>2</sub> in Dough: In order to estimate how much of the total acidity was due to CO<sub>2</sub>, samples of dough were prepared and handled in the same way as for total acidity, but the extracts were aspirated for 3 hours with CO<sub>2</sub>-free air, 0.5 cc. of neutral formaldehyde having been added to prevent yeast activity during the aeration. Tables X and XI, and Figures 4 and 5 show the degree to which CO<sub>2</sub> influenced the values for total acidity.



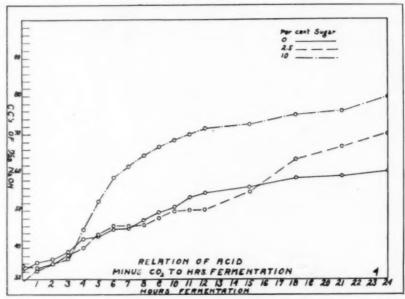


Fig. 3. Relation of total acidity to hours of fermentation.

Fig. 4. Relation of acid minus CO2 to hours of fermentation.

TABLE VIII ALCOHOL CONTENT EXPRESSED AS PER CENT BY WEIGHT PER 100 GRAMS OF FLOUR

Farmentation		Per cent sugar in	doughs
Fermentation time	0	21/2	10
Hrs.	Alcohol	Alcohol	Alcohol
	P.ct.	P.ct.	P.ct.
0	0.00	0.00	0.00
0	0.35	0.75	0.75
. 2 3 4 5 6 7 8	1.12	1.16	1.00
3	1.40	1.38	1.25
4	1.71	1.94	1.52
5	1.88	2.09	2.18
6	1.96	2.15	2.50
7	1.84	2.21	2.32
8	1.85	2.38	2.40
	1.87	2.43	
10	-	-	
11			
12	2.11	2.78	3.70
15	2.36		4.25
18	2.82	3.00	4.62
21	2.78	3.28	5.10
24	2.79	3.50	
26			5.11

TABLE IX TOTAL ACIDITY EXPRESSED AS N/28 NAOH PER 100 GRAMS OF FLOUR

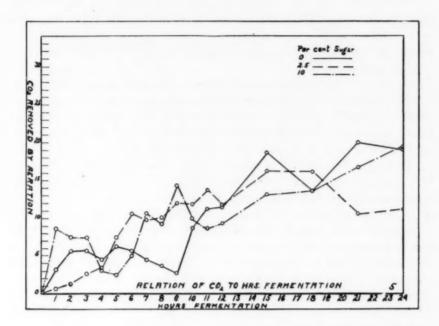
Fermentation		Per cent suga	ır
time	0	21/2	10
	N/28 NaOH	N/28 NaOH	N/28 NaOH
Hrs.	cc.	· cc.	cc.
0	34.37	35.62	31.87
	39.37	35.62	43.12
2	43.12	37.50	43.75
1 2 3 4 5	45.00	41.25	45.00
4	46.87	45.62	48.12
5	48.75	51.25	55.00
6	49.37	55.62	63.75
6 7 8 9	50.00	55.00	72.50
8	51.25	56.25	73.75
9	52.50	60.00	81.25
10	60.00	61.25	78.75
11	65.00	63.75	78.75
12	66.25	63.75	81.25
13	64.37	71.25	79.37
14	69.37	72.12	85.00
15	75.00	71.55	85.62
16	73.10	77.50	86.25
17	73.10	81.25	87.50
18	71.47	80.00	88.75
20		78.75	90.00
21	79.37	77.50	93.12
23			
24	80.25	81.25	98.75

TABLE X
ACIDITY LESS CO<sub>2</sub> Removed by 3 Hours Aerating with CO<sub>2</sub>-Free Air

Fermentation time		Per cent sug	ar
	. 0	21	10
	N/28 NaOH	N/28 NaOH	N/28 NaOH
Hrs.	cc.	cc.	cc.
0	34.37	35.62	31.87
	36.25	35.00	34.37
2	37.50	36.35	36.25
3	39.37	38.75	37.50
4	42.50	41.25	45.00
1 2 3 4 5 6 7 8	43.12	43.75	52.50
6	45.00	45.00	58.75
7	45.62	45.92	61.87
8	47.50	46.25	64.50
9	49.82	48.00	66.87
10	51.25	49.37	68.75
11	53.75	50.00	70.00
12	54.75	51.87	71.87
13			
14	-		
14 15	56.25	55.00	72.50
16 17			-
17		-	
18	. 58.75	63.75	75.00
20			-
21	59.37	66.87	76.25
24	61.25	70.12	79.37

TABLE XI PROGRESSIVE DEVELOPMENT OF CO2 DURING FERMENTATION, EXPRESSED AS CC. OF N/28 NAOH PER 100 Grams of Flour

Fermentation time		Per cent suga	r
	0	21	10
	N/28 NaOH	N/28 NaOH	OH N/28 NaOH
Hrs.	cc.	cc.	cc.
0	0.00	0.00	0.00
	3.12	0.62	8.75
2	5.62	1.25	7.50
1 2 3 4 5 6 7 8	5.63	2.50	7.50
4	4.37	3.37	3.12
5	6.25	7.59	2.50
6	5.63	10.62	5.00
7	4.38	9.08	10.36
8	3.75	10.00	9.25
9	2.68	12.00	14.38
10	8.75	11.88	10.00
11	11.25	13.75	8.75
12	11.30	11.88	9.38
13			-
14		-	
15	18.75	16.25	13.12
16		-	
17		-	
18	13.72	16.25	13.75
20			
21	20.00	10.63	16.87
24	19.00	11.13	19.33



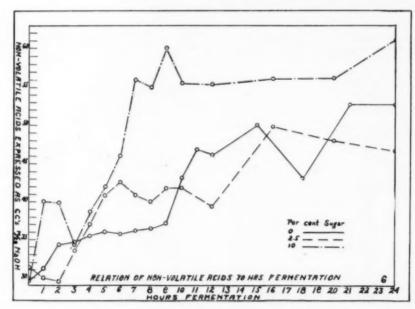


Fig. 5. Relation of CO<sub>2</sub> to hours of fermentation. Fig. 6. Relation of non-volatile acids to hours of fermentation.

DETERMINATION OF VOLATILE AND NON-VOLATILE ACIDITY IN DOUGH: Johnson (1925) found that lactic and acetic acids were the only organic acids produced in appreciable quantities during bread dough fermentation, lactic acid constituting about 75% of the total organic acid content.

Lactic acid being non-volatile, in contrast with acetic acid, it was considered desirable to comparatively study the progressive development of volatile and non-volatile acidity in the doughs of varying initial sugar content as indicative of the comparative rates of development of lactic and acetic acids under these conditions.

The apparatus used was essentially the one described by Plimmer (1917) for the estimation of amide N in the products of hydrolysis of proteins. The dough was mechanically disintegrated in 300 cc. of water in the soda-fountain mixer, centrifuged, and the clear extract evaporated to dryness in the distilling flask at 55–60° C. and 28–30 mm. pressure, the distillate being caught in standard alkali; 50 cc. of water were added to the residue in the distilling flask and the process repeated. Three such distillations were found to be sufficient to remove all volatile acids. Values for volatile and non-volatile acidity in the experimental doughs are shown in Tables XII and XIII, respectively, and in Figures 6 and 7.

TABLE XII

Non-volatile Acids in Terms of Alkali Required to Neutralize 100 Cc. of Dough Suspension, Expressed as Cc. of N/28 NaOH per 100 Grams of Flour

Fermentation		Per cent sugar		
time	0	21	10	
	N/28 NaOH	N/28 NaOH	N/28 NaOH	
Hrs.	cc.	cc.	cc.	
0	29.67	31.32	29.47	
1	31.17	29.92	39.92	
2	34.22	29.50	39.25	
3	34.60	33.45	34.10	
4	35.37	36.92	38.72	
5	36.05	40.85	41.80	
6	35.87	42.72	45.85	
2 3 4 5 6 7 8	36.10	40.90	56.20	
8	36.55	39.85	55.05	
9	37.10	41.80	.60.85	
10	43.10	41.75	55.65	
11	46.80			
12	46.15	39.35	55.45	
13				
14		-		
15	49.50			
16		48.70	56.11	
17				
18	43.17		-	
20		48.05	56.30	
21	52.97			
24	52.85	46.65	61.15	

TABLE XIII

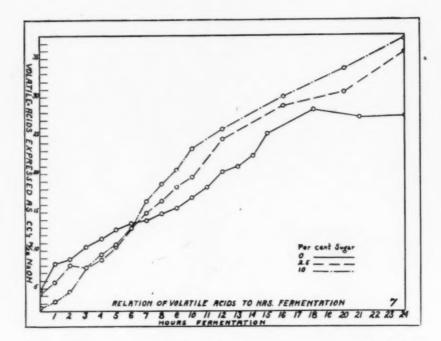
Volatile Acids in Terms of Alkali Required to Neutralize 100 Cc. of Dough Suspension, Expressed as N/28 NaOH per 100 Grams of Flour

Fermentation		Per cent suga	r	
time	0	21	10	
	N/28 NaOH	N/28 NaOH	N/28 NaOH	
Hrs.	cc.	cc.	cc.	
0	4.7	4.3	2.5	
	8.2	5.7	3.2	
2	8.9	8.0	4.5	
3	10.4	7.8	7.9	
4	11.5	8.7	9.4	
5	12.7	10.4	10.7	
6	13.5	12.9	12.9	
1 2 3 4 5 6 7 8	13.9 .	14.1	16.3	
8	14.7	16.4	18.7	
9	15.4	18.2	20.4	
10	16.9	19.5	23.1	
11	18.2		-	
12	20.1	24.4	25.8	
13	20.9			
14	22.3		-	
15	25.1		-	
16	-	28.8	30.01	
17	-	-		
18	28.3	MODERNING.		
20		30.7	33.7	
21	27.2		-	
24	27.4	35.7	37.6	

TABLE XIV

LOAF VOLUME, FERMENTATION TIME AND PERCENTAGE OF SUGAR

Fermentation time		Per cent sug	ar
cinie	. 0	21	10
Hrs.	cc.	cc.	cc.
0	402	387	338
1	492	508	512
2	467	538	529
3	355	516	608
4	357	437	612
4 5	340	352	575
6		348	560
7		350	525
8	-		510
9	-		464
10	-		430
11	Section 1999		382
12			338



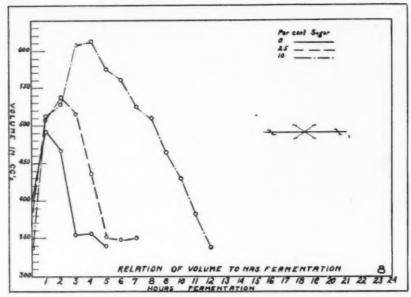


Fig. 7. Relation of volatile acids to hours of fermentation.

Fig. 8. Relation of volume to hours of fermentation.

#### Discussion

In the three series of bread photographs shown in Figures 9 and 10 the numbers on the loaves indicate hours of dough fermentation, to which in each case may be added an additional hour (55 minutes) of fermentation during pan proof. A mere inspection of these photo-

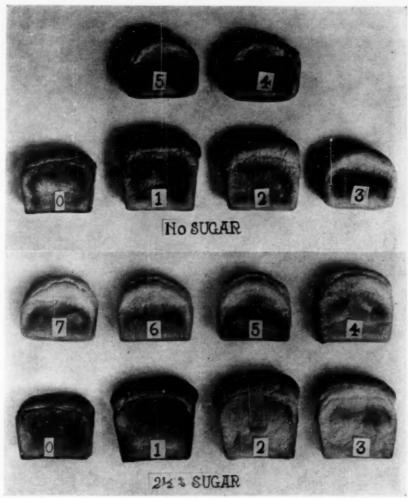


Fig. 9. Upper half—loaf volume and fermentation time without sugar; lower half—loaf volume and fermentation time with normal sugar content.

graphs is all that is necessary to establish the general idea that fermentation tolerance is dependent upon a maintained supply of fermentable sugars in the dough. In the "no sugar" series, the supply of fermentable sugar was of course restricted to the quantity produced by the natural diastatic enzymes of the flour. Here it is apparent that the limit of tolerance was approximately 2 hours. When  $2\frac{1}{2}\%$  sugar was included in the mixture of dough ingredients, the period of tolerance was in excess of 3 hours, while in the 10% sugar series tolerance was increased to approximately 9 hours.

These baking results clearly demonstrate that under the conditions and specifications of the A. A. C. C. basic procedure, resistivity to prolonged yeast fermentation is predominatingly a function of the gas-producing agency, i.e., maintenance of an adequate supply of fer-

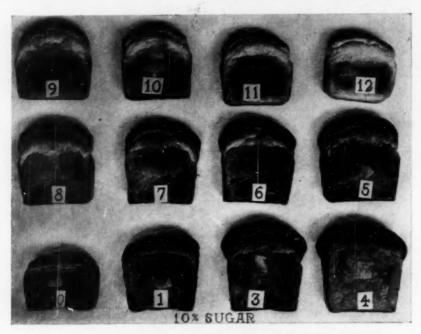


Fig. 10. Showing influence of 10% of sugar on fermentation time and loaf volume.

mentable sugar in the dough. It, therefore, becomes perfectly clear that differences in fermentation tolerance, as measured by the A. A. C. C. basic procedure (in which all flours receive the same sugar dosage as well as the same treatment in all other respects), simply reflect variations among the natural diastatic, or "saccharogenic," or "gassing power" of the flours.

The standard A. A. C. C. basic procedure may well be regarded as primarily a diastatic activity test, with depth of crust color as perhaps the most important diagnostic symptom of fermentation tolerance. Those test loaf properties that reflect the character of the gas-retaining agency, i.e., the gluten, have diagnostic value only when there is

definite assurance that there has been an adequately maintained gas production.

Let us assume that a flour baked by the standard basic procedure gave a test loaf resembling the 4 hour loaf in the  $2\frac{1}{2}\%$  sugar series (Figures 9 and 10). This loaf shows characteristic symptoms of "age," and on account of its small volume and its tendency toward "shell top" might be condemned as having poor gluten quality. Yet when 10% sugar was used the same flour withstood as much as 7 and 8 hours of fermentation (7 and 8 hour loaves, Figure 10) without showing symptoms either of "age" or of poor gluten quality.

There is little doubt that these so-called "age symptoms" have been frequently misinterpreted in the laboratory testing of wheat and flour quality. Flours have been and are often condemned as "weak," implying inferiority as to their gluten or *structural* properties, when in reality they may have excellent gluten properties but were baked under conditions of insufficiently maintained gas production, due either to low diastatic power, to insufficient sugar in the formula, to too high a fermentation temperature, to too much yeast, or to any combination of these factors. Strength and "gassing power" of flour should be regarded as separate and distinct factors, as have been suggested by Wood (1907), Fisher and Halton (1929), Jørgensen (1931), and others. Contrary to the beliefs of Jørgensen (1931), however, the standard A. A. C. C. baking test, with its basic and supplementary procedures, affords ample and reliable means for distinguishing between the two.

The tables and figures present detailed information and data regarding the rates at which the more conspicuous by-products of alcoholic fermentation (H-ions, alcohol, total acidity, volatile and non-volatile acidity) develop in a bread dough over a time range of 0 to 24 hours, where only simple ingredients (yeast, sugar, and salt) are used. They show comparative differences in the rates at which these by-products accumulate under conditions (a) where active alcoholic fermentation is restricted by entirely withholding sugar from the dough mixture, (b) where the period of active fermentation is increased by using  $2\frac{1}{2}\%$  sugar in the dough, and (c) where active fermentation is maintained over an unusually long period (about 10 hours) of time by the initial incorporation of 10% sugar in the dough mixture.

As would be anticipated, the progressive development of acids and of alcohol was greatest in the 10% sugar series. However, these products also continued to gradually increase with time in the other series long after active fermentation had ceased. The doughs in the "no sugar" series produced almost as much volatile (acetic acid)

acidity over most of the 24 hour period as did the doughs of the 10% sugar series. This may mean that organisms other than yeast were largely responsible for the production of acetic acid.

Figures 3 and 4 show that a considerable portion of the total acidity is due to carbon dioxide, which is in agreement with Johnson (1925). Data on non-volatile and volatile acidity indicate that the greater portion of the organic acids produced is lactic acid, which also is in conformity with the findings of Johnson (1925).

An inspection of Figure 1, depicting the progressive development of H-ions throughout the 24 hour period, with doughs of different initial sugar concentration, reveals a situation that perhaps is not what might reasonably be expected in the light of progressive changes in acidity, and of previous findings by Johnson and Bailey (1924) and by Johnson (1925). Progressive changes in pH appear to have followed essentially the same course regardless of initial sugar content or of the duration of the period of active alcoholic fermentation. In all three cases there was a progressive and steady decrease in pH for the first 7 hours, similar to that reported by Bailey and Sherwood (1923), who studied the march of H-ions in commercial doughs over a 6 hour period. Figure 1 shows that all three doughs reached a pH of approximately 5 in 7 hours and each dough maintained this value throughout the remainder of the 24 hour period.

These data certainly do not conform to what might be expected from the values for the titratable acidity, where the 10% sugar doughs showed a substantially greater rate of increase in acidity than did the other two series.

In reaching a value of approximately pH 5.0 in 7 hours, and maintaining this value almost constant for the remaining 17 hours, these doughs differ from the cracker doughs studied by Johnson and Bailey (1924), where the pH steadily decreased for 24 hours, ultimately reaching values slightly below 4.0. Neither do these pH values conform to the findings of Johnson (1925) with flour-water suspensions containing yeast, in which a pH of 3.27 was reached in 24 hours.

The true reasons for these H-ion results are not clear to the writers, although it must be recognized that in physical properties a bread dough is strictly comparable neither to a cracker dough nor to a flour-water suspension, being much more hydrated than the former and much less hydrated than the latter. At any rate, under the conditions herein reported, the pH in all three series reached a value of around 5.0 in 7 hours, after which there appears to have been a decided "buffer action" that prevented a further decrease in pH regardless of constant increasing titratable acidity.

In a foregoing portion of this report, mention has been made of the widespread belief that fermentation tolerance is to a substantial degree associated with the manner in which the gluten gas-retaining structure of the dough is affected by fermentation by-products, notably acids, alcohol, and hydrogen-ions, and by proteolytic enzymes. The findings herein reported afford no basis for a substantiation of this belief as applied to sound normal flour fermented and baked with the simple ingredients, yeast, sugar and salt. Whatever influence these factors may have exerted was so slight as to be completely overshadowed by the importance of a maintained supply of fermentable sugars.

One may consider the nature of the curve showing the relation between loaf volume and fermentation time for the 10% sugar dough (Figure 8) in the light of the data showing progressive increases in fermentation by-products (Figures 1 to 7). The photographs of the loaves shown in Figures 9 and 10 show clearly that when the limit of fermentation tolerance is exceeded, symptoms of age are substantially the same in all three series, regardless of the number of elapsed hours of fermentation. Comparing the 10 or 11 hour loaf in the 10% sugar series with the 4 hour loaf in the  $2\frac{1}{2}\%$  sugar series it will be observed that the loaf characteristics and "age" symptoms are for all practical purposes identical. The analytical data show, however, that the 10 and 11 hour doughs were much higher in acid, H-ion, and alcohol concentration than the 4 hour dough; yet the loaf from the 11 hour dough shows no more evidence of weakened structural properties than does the loaf from the 4 hour dough.

Figure 8, depicting the relationship of loaf volume to fermentation time for each of the 3 sugar concentrations, shows that the 3 curves are about identical in shape, slope, and general conformity, despite the greater acid and alcohol concentrations reached in the high sugar doughs. The curves bear no apparent relationship to the H-ion, acidity or alcohol curves. The curves differ only in magnitude, and show that the greater sugar concentrations not only extended the fermentation tolerance of the dough but also provided greater maximum loaf volume. This is in agreement with the findings of Wood (1907), who concluded that loaf volume depends largely upon the amount of available sugar present in the later stages of fermentation. Others, too numerous to mention, have had similar experiences. The conclusion that pH is an insignificant factor in baking supports the findings of Fisher and Halton (1929).

This report and discussion is intended in no sense to imply that the factor of gas production is the all-important agency in bread-making, or to in any sense minimize the importance of the gluten. It is concerned primarily with *resistivity* to *fermentation*, when baking by the

straight dough method, using only the simple leavening and conditioning ingredients, yeast, sugar and salt, as is done in the standard A. A. C. C. basic procedure. Increasing the available sugar content will certainly not remedy inferior baking characteristics that are due to deficiency in either the quantity or the character of the gluten, even though it may add appreciably to loaf volume. The loaf may be superior or decidedly inferior, as the gluten properties will permit, but whatever the type of loaf, the fermentation tolerance, i.e., the time range over which that particular type can be produced, will be governed predominantly if not for all practical purposes exclusively-by the extent to which gas production is maintained. This may be controlled by regulating the supply of fermentable sugar. Progressive deterioration of the gluten by those by-products of fermentation that are frequently considered to operate toward that end (acid, H-ions, alcohol, and proteolytic enzymes) is a negligible factor in type of baking to which this report is confined.

Applications of these results to commercial baking are obvious. They show that in straight dough baking fermentation tolerance may be entirely governed by the amount of sugar used in the dough batch, provided no unsound flour, sprouted wheat flour, or diastatic malt (containing proteases) is used. They show why the sponge and dough method is more "fool-proof" than the straight dough method, for the sponge and dough method provides for the addition of sugar only at the dough time, and just before the proofing period, thereby insuring an abundance of fermentable sugars during the rise in the pan. This also insures a good crust color, provided oven conditions are reasonably correct. Variations in sponge time are of no serious consequence, under ordinary bake-shop conditions, unless diastatic malt or similar preparations are used in the sponge. This viewpoint is supported by the experiments of Harrel (1926).

## Summary and Conclusions

- 1. A reliable technique has been developed for the estimation of alcohol content of bread dough.
- 2. Using the technique of the standard A. A. C. C. basic procedure for the preparation of doughs, studies of progressive changes in H-ion concentration, alcohol content, total acidity, and acidity due to CO<sub>2</sub> and to volatile and non-volatile organic acids in doughs with initial sugar concentrations varying from sugar deficiency to sugar excess were made over a 24 hour range of fermentation time. An effort was made to correlate the findings with actual baking tests involving corresponding intervals and durations of fermentation time.

3. In the "no sugar" dough series, the period of active alcoholic fermentation was approximately 2 hours, it was nearly 4 hours in the  $2\frac{1}{2}\%$  sugar dough, and 9–10 hours in the 10% sugar dough.

4. Progressive changes in H-ion concentration followed essentially the same course in all three doughs, regardless of the duration of the period of active fermentation. The pH values all decreased steadily to approximately 5.0 in 7 hours, remaining constant at 4.9-5.0 during the remainder of the 24 hour period.

5. The 10% sugar doughs reached substantially higher concentrations of titratable acidity and alcohol, respectively, during the 24 hour period than did the "no sugar" and the  $2\frac{1}{2}\%$  sugar doughs. In all doughs, however, the concentrations of alcohol and of acids steadily increased even after active alcoholic fermentation had apparently ceased.

6. The greater portion of the organic acids produced was nonvolatile (probably lactic) acid.

7. In straight dough baking, and using only the simple ingredients, yeast, sugar, and salt, fermentation tolerance, or resistivity to fermentation alone, depends predominantly—and for all practical purposes exclusively-upon maintained gas production, as governed by the supply of fermentable sugar. These studies afford no indication that proteolytic enzymes and the more conspicuous by-products of fermentation, i.e., H-ions, acidity and alcohol, effect any serious changes that might be regarded as in the nature of "gluten degradation" within any time range that is likely to be employed in bread production.

8. The factor of gas production (sugar and diastatic action) is for all practical considerations independent of the gas retention factor (gluten) under normal baking conditions, where only simple ingredients are employed, as in the type of procedure specified in the standard A. A. C. C. experimental baking test.

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# METALLIC SELENIUM AS A CATALYST IN KJELDAHL DIGESTIONS

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The recent interest in the use of selenium and some of its derivatives as a catalytic agent in Kjeldahl digestions has stimulated investigations with respect to reducing the time necessary for such determinations. However, in all investigations reported to date, there seems to be a desire to omit the use of mercury when selenium is used, presumably because the use of mercury makes the use of a precipitant before distillation necessary.

It occurred to us that the use of mercury and selenium together might prove advisable. Inasmuch as Rich (Cereal Chem. 9: 118–121, 1932) found that the use of selenium oxchloride and copper together accelerated the digestion reaction more than when either was used alone, experiments to this effect have been carried out in this laboratory with encouraging results.

550 watt heaters, in every case cold at the start of the digestion, were used. 20 cc. H<sub>2</sub>SO<sub>4</sub> and 8 gms. of a flux consisting of 90 parts of Na<sub>2</sub>SO<sub>4</sub>, 7 parts of HgSO<sub>4</sub>, 1½ parts of CuSO<sub>4</sub>, and 1½ parts of powdered metallic selenium by weight, were added to each one gram sample to be digested with the following results:

	Time of digestion							
Material	15 min. Per cent protein	20 min. Per cent protein	25 min. Per cent protein	30 min. Per cent protein	35 min. Per cent protein			
Flour	(N × 5.7) 10.88	(N × 5.7) 10.93	(N × 5.7) 10.90	(N × 5.7) 10.90	(N × 5.7 10.88			
Flour	13.95	13.90	13.98	13.95	13.95			
Flour	14.00	14.00	13.97	14.00	14.00			
Wheat	11.53	11.55	11.50	11.58	11.55			
Wheat	17.53	17.50	17.50	17.55	17.53			
Wheat	12.60	12.55	12.55	12.58	12.55			

On the basis of the results as well as from other work, this laboratory has adopted the above mentioned flux as a catalyst, as it is believed that digestion is complete in 15 to 20 minutes, thus making it possible to make a complete nitrogen determination in less than an hour. The cost of selenium used is less than a quarter of a cent per determination. The saving in current consumption alone more than offsets this cost.

# EFFECTS OF THE AMOUNT AND KIND OF BLEACH USED ON FLOUR IN RELATION TO ITS AGING

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(Received for publication January 6, 1932)

#### Introduction

The chemical and physical changes which take place when bleached or chemically treated flours are placed in storage have not been clearly determined. It is generally known that flour which has been stored for any length of time may show a decided change in both chemical and physical properties.

The coloring matter of flour is considered to consist of carotin and xanthophyll, the latter being present in the bran particles. Carotin is

readily decolorized by certain oxidizing agents.

Color is a variable property in flour, and we may consider it as being permanent only after the changes which come from aging or bleaching. The color of unbleached flour is generally taken as one index of quality, as it may indicate something of the variety of wheat from which the flour has been milled, as well as the grade of the flour.

While, in a general way, the changes in color which take place when flour is aged may be described, up to the present time it is not possible to measure them with the same degree of accuracy as is the case in the determination of other flour characteristics.

The early conceptions of the significance of flour bleaching varied considerably from our present views. Mill superintendents and millers did not agree on the benefits of bleaching, which today is almost universally accepted as essential to good milling. The propriety of the use, under the Federal Food and Drugs Act, of nitrogen peroxide as a bleaching agent for flour, was questioned by the United States Government. After hearings had been held by the Board of Food and Drug Inspection, the matter was finally submitted to the courts. In the end, flour bleaching was permitted, the flour containers to be marked "Bleached." The last decision of the Government (Service and Regulatory Announcement 26, Bureau of Chemistry No. 26, page 21) permits bleaching if the process does not introduce into the flour such a quantity of the bleaching agent as may be injurious to health.

<sup>&</sup>lt;sup>1</sup> Abstract of a thesis submitted to the faculty of Kansas State College in partial fulfilment of the requirements for the degree of master of science.

## Principal Chemical Bleaching Agents

The four chemical substances or the reagents which are employed for the bleaching of flour at the present are: Nitrogen peroxide, benzoyl peroxide, nitrogen-trichloride, and chlorine. Chlorine, as such, is seldom used for the commercial treatment of flour. It is usually used in admixture with nitrosyl chloride, this mixture containing 99.5% of chlorine and 0.5% of nitrosyl chloride, to which the trade name of Beta Chlora is applied.

Nitrogen peroxide is the oldest of these flour bleaching reagents. In combination with carotin ( $C_{40}H_{56}$ ) it forms a colorless compound of unknown composition. When nitrogen peroxide is applied to the flour it acts not only upon the carotin, but it also reacts with the water present in the flour. This reaction with the water takes place according to the equation:

$$2NO_2 + H_2O \rightarrow HNO_2 + HNO_3$$
Nitrous
acid
Nitric
acid

Benzoyl peroxide is a white crystalline product which has been used for many years in the bleaching of fatty oils. By itself it is somewhat explosive. For this reason it is properly blended with carrying agents and ground to a powder for flour bleaching purposes. The well known bleaching agent "Novadelox," which has been used extensively in recent years, is said to contain 25% benzoyl peroxide and 75% acid calcium phosphate. Benzoyl peroxide easily releases its oxygen thereby changing into benzoic acid, while the oxygen liberated reacts with the coloring matter of the flour. One property that makes benzoyl peroxide a very desirable bleaching reagent is its solubility in fats. Carotin is likewise soluble in fats, and is probably present in the wheat associated with the fats. It is evident then that a bleaching reagent which is soluble in the fatty constituents of the flour must be more active than one that is not. The reaction of benzoyl peroxide with water is:

$$(C_6H_8CO)_2O_2 + H_2O \rightarrow 2C_6H_8COOH + O_2$$
  
Benzoyl peroxide Benzoic acid

When chlorine is applied to flour it not only unites with the carotin, but also with the other constituents, particularly the unsaturated fats. The fat in a chlorine bleached flour will thus contain a larger proportion of the chlorine present than that of a corresponding kind of flour which has not been bleached with chlorine. This is of importance in the analytical detection of the bleaching of flour with chlorine. The action of chlorine in whitening flour is undoubtedly not

due wholly to the action of chlorine per se, but to its oxidizing capacity, through its reaction with the water of the flour, forming hypochlorous acid:

 $Cl_2 + H_2O \rightleftharpoons HClO + HCl$ 

When nitrogen-trichloride, commercially known as "Agene," is applied to flour, changes in chemical composition are difficult to detect. The ash content, wet and dry gluten content, the percentage of total of water soluble proteins, as well as the titratable acidity of the flour are not altered in so far as can be determined by ordinary laboratory methods. There does, however, appear to be an increase in the hydrogen-ion concentration when the dosage is in excess of 2 to 3 grams per barrel. Even in such extreme cases, however, the increase in pH is slight. The color of the flour is, of course, markedly changed by its use; an aging effect is also to be noticed.

Nitrogen-trichloride gas is formed according to the following equation:

 $3Cl_2 + 3H_2O \rightarrow 3HOCl + 3HCl$  $3HOCl + NH_4Cl \rightarrow NCl_3 + 3H_2O - HCl$ 

The maturing of flour with chlorine bleach is used extensively. It appears that optimum results are approached in a very limited time. By the use of this bleaching agent the gluten of the flour is so modified that flours properly bleached should produce maximum baking characteristics shortly after bleaching.

# Purpose of the Investigation

It is presumed that flours which have been bleached and placed in storage undergo both chemical and physical changes. Just what effect different degrees of treatment with the usual bleaching reagents have on stored flour, as reflected in baking characteristics, does not appear to have been extensively studied, and it is the purpose of this investigation to accumulate certain data to give information on these points.

#### Method of Procedure

FLOUR. As material for this study, 95% standard patent flours were milled from wheats of the 1929 and 1930 crops of hard red winter wheat. Especial attention was given to the selection of the wheat so that each lot of flour would have approximately the same composition. The wheats used were the varieties Kanred and Blackhull. The Kanred wheat of the crop year 1929 had been stored at the Department of Milling Industry prior to milling. The Kanred and Blackhull wheats of the 1930 crop were purchased at the start of the 1930 harvest. Kanred was a combine harvested wheat. Sufficient wheat

was milled of the Kanred type to furnish fourteen 48-pound sacks of flour. However, only sufficient Blackhull wheat was milled to furnish approximately four 48-pound sacks of flour.

The flour was not bleached at the time of milling, but was within a period of 24 hours by the use of carefully controlled equipment.

CHEMICAL ANALYSIS OF THE FLOURS

	Kanred 1929	Kanred 1930	Blackhull 1930	
Per cent ash	0.420	0.430	0.430	
Per cent protein	11.20	10.65	10.48	
Per cent moisture	13.00	12.90	12.30	

BLEACHING AGENTS. The following commercial bleaches were used: Nitrogen-trichloride, benzoyl peroxide, and Beta Chlora (chlorine plus nitrosvl chloride).

The flours were bleached according to the following rates of application:  $\frac{1}{3}$  bleach,  $\frac{2}{3}$  bleach,  $\frac{3}{3}$  (full bleach), and  $\frac{6}{3}$  bleach (double). The exact amount of bleaching agent added in each case was calculated on the following basis: benzoyl peroxide, 1 pound to 40 barrels of flour; nitrogen-trichloride, 4.4 gms. of gas per barrel; chlorine (Beta Chlora), 3/4 ounce of gas per barrel of flour. The amounts specified are for a full bleach, other amounts were added in proportion.

#### Methods

LABORATORY TESTS. The Pekar test was made immediately after the flours were bleached to determine the relative extent of the bleaching. This test showed a range in color from a decided yellowish tint to the usual white color. In addition to the routine ash, protein, and moisture determinations, further analyses were made from time to time with respect to the hydrogen-ion concentration of the flours and the viscosity of flour in water suspensions in order to ascertain whether changes in composition during storage were taking place which could be detected by such methods.

BAKING TESTS. Four methods were used in making the baking tests; a sponge and dough method; a two hour, short fermentation, straight dough method; the Swanson 2 mechanical modification method; and the potassium bromate differental method. All loaves were baked in duplicate except when the potassium bromate differential method was used (Werner and Herman).3

<sup>&</sup>lt;sup>2</sup> C. O. Swanson, The mechanical method of modification of dough. Cereal Chem. 5: 375-385,

<sup>1928.
&</sup>lt;sup>2</sup> E. E. Werner and R. S. Herman, The differential baking test. Northwestern Miller and American Baker 5: 12-16, 1928.

In interpreting the data a difference of less than two points in the score of crumb color and texture, and a difference of 30 cc. on loaf volume are not considered significant.

## **Experimental Data**

### RESULTS BY SPONGE AND DOUGH METHOD

### Benzoyl Peroxide Bleached Flours

The results of the tests made with the benzoyl peroxide bleached, 1929 crop, Kanred flours as baked by the sponge and dough method, at five different intervals, are shown in Table I. The results demonstrate that the use of benzoyl peroxide, even in very large amounts, did not

TABLE I

RESULTS OF BAKING TESTS ON BENZOYL PEROXIDE BLEACHED, 1929 CROP, KANRED FLOUR BAKED BY SPONGE AND DOUGH METHOD

Loaf number	Amount of	Date of baking						
	bleach	June 20	June 27	July 7	July 23	Aug. 13	Dec. 2	
		Loaf ve	olumes in c	ubic centi	meters			
1	Unbleached	1565	1550	1545	1553	1520	1520	
2	1/3 bleach	1585	1575	1570	1575	1623	1535	
3	2/3 bleach	1570	1588	1575	1565	1577	1533	
2 3 4 5	3/3 bleach	1605	1570	1585	1575	1610	1568	
5	6/3 bleach	1567	1545	1580	1585	1575	1530	
		C	olor of crui	mb-score				
1	Unbleached	94	95	96	97	97	97	
2 3	1/3 bleach	95	96	97	97	98	96	
3	2/3 bleach	96	96	98	98	98	96	
4	3/3 bleach	97	97	98	98	98	97	
5	6/3 bleach	98	98	99	99	98	98	
		Te	xture of cri	umb-score				
1	Unbleached	94	96	97	98	98	98	
2	1/3 bleach	96	97	98	98	98	98	
2 3 4 5	2/3 bleach	96	96	98	98	98	98	
4	3/3 bleach	97	98	98	98	97	96	
5	6/3 bleach	97	96	98	98	97	99	

impair the baking quality of the flour in any way. On the other hand, there was a significant improvement in color and texture of crumb with increasing amounts of the bleach for a period of about a week, after which all loaf characteristics on all subsequent bakings were practically identical. The slight decrease in loaf volume in the loaves baked on December 22 is probably due to an error in ascertaining the proper water absorption.

The results obtained with the Kanred flour of the 1930 crop bleached with benzoyl peroxide are given in Table II. The results are closely the same as those recorded for the bakings with the flour of the 1929 crop. Loaf volume is fairly constant, and there is some improvement

TABLE II

RESULTS OF BAKING TESTS ON BENZOYL PEROXIDE BLEACHED, 1930 CROP, KANRED FLOUR, BAKED BY THE SPONGE AND DOUGH METHOD

Loaf number	Amount of bleach	Date of baking					
		Aug. 16	Aug. 20	Sept. 9	Oct. 13	Jan. 22	
		Loaf volume	e in cubic cer	ntimeters			
1	Unbleached	1545	1505	1510	1515	1520	
2	1/3 bleach	1515	1500	1515	1508	1490	
3	2/3 bleach	1595	1490	1495	1500	1545	
2 3 4 5	3/3 bleach	1490	1505	1510	1505	1530	
5	6/3 bleach	1485	1473	1480	1490	1597	
		Color	of crumb-sc	ore			
1	Unbleached	95	95	96	97	98	
2	1/3 bleach	95	96	96	97	97	
2 3 4 5	2/3 bleach	95	96	96	97	97	
4	3/3 bleach	96	96	96	98	97	
5	6/3 bleach	97	97	98	98	98	
		Textur	e of crumb-s	core			
1	Unbleached	95	96	98	98	98	
2	1/3 bleach	95	97	98	98	98	
2 3 4 5	2/3 bleach	95	97	97	98	98	
4	3/3 bleach	95	97	97	97	98	
5	6/3 bleach	96	97	98	97	98	

in color of crumb with an increase in the amount of bleach. In this series, likewise, there was no apparent injury to baking characteristics with increasing amounts of benzoyl peroxide. Such differences as are apparent in the early stages of the investigation are not apparent as aging of the flour progresses. Photographs of the loaves, baked at the start of the experiment (August 16) are shown as Figure 1.

# Nitrogen-Trichloride Bleached Flours

The results obtained with the flour milled from the 1929 and 1930 crops of Kanred wheat, which were bleached with nitrogen-trichloride, and baked by the sponge and dough method, are shown in Tables III and IV. The baking results would indicate that the baking quality of the flour was not impaired in any way by the use of double the amount of nitrogen-trichloride necessary to effect a full bleach. There was a gradual improvement in color of crumb with increasing amounts of the

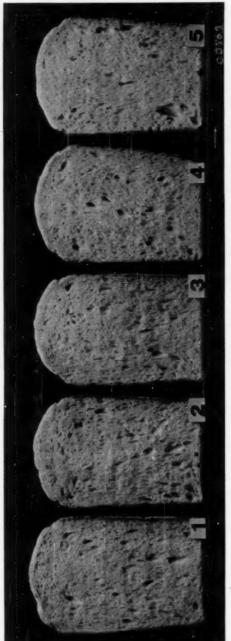


Fig. 1. Loaves baked by sponge and dough method from benzoyl peroxide bleached, Kanred wheat flour, crop 1930.
Fig. 1. Loaves baked by sponge and dough method from benzoyl peroxide bleached, Kanred wheat flour, crop 1930.

bleach soon after application, as recorded by the first day's bake. However, no significant changes in loaf characteristics were to be noted with subsequent aging in storage of the bleached flour. The slight decrease in loaf volume (1929 crop flour) obtained on December 5 is probably due to an error in measuring absorption rather than to an aging effect.

TABLE III

RESULTS OF BAKING TESTS WITH NITROGEN-TRICHLORIDE BLEACHED, 1929 CROP, KANRED FLOUR, BAKED BY THE SPONGE AND DOUGH METHOD

Loaf number	Amount of bleach	Date of baking					
		June 22	June 29	July 9	July 26	Dec. 5	
		Loaf volume	es in cubic ce	entimeters			
1	Unbleached	1560	1550	1545	1555	1550	
2	1/3 bleach	1580	1570	1588	1615	1550	
2 3 4 5	2/3 bleach	1610	1600	1590	1605	1555	
4	3/3 bleach	1596	1605	1575	1590	1528	
5	6/3 bleach	1623	1605	1595	1590	1545	
		Color	of crumb-sc	ore			
1	Unbleached	94	95	97	97	97	
2	1/3 bleach	95	97	98	98	98	
2 3 4 5	2/3 bleach	96	98	98	98	98	
4	3/3 bleach	96	98	98	98	98	
5	6/3 bleach	98	99	99	99	99	
		Textu	ure of loaf-sco	ore			
1	Unbleached	96	96	98	98	97	
2	1/3 bleach	98	97	98	98	98	
2 3 4 5	2/3 bleach	98	97	98	98	98	
4	3/3 bleach	98	98	98	98	98	
5	6/3 bleach	97	97	98	98	98	

The baking tests with the flour from the 1930 crop of Kanred wheat, which was bleached with nitrogen-trichloride and baked by the sponge and dough method, are given in Table IV. The results obtained on all bakings with respect to loaf volume, color of crumb or texture of crumb, were closely the same over the entire period of the investigation.

From the data presented it would appear that nitrogen-trichloride so conditions flour that optimum baking results are made possible almost immediately. This beneficial effect can be noted by comparing the data obtained with the unbleached flour with that of the full and double bleached flours. The color and texture of the crumb of the bleached flour loaves did not improve materially with the aging of the flour. At no time did an excess of nitrogen-trichloride appear to

impair the baking quality of the flour. Photographs of the loaves from the 1930 crop series of tests, baked on August 21, 1931, are shown as Figure 2.

TABLE IV

RESULTS OF BAKING TESTS WITH NITROGEN-TRICHLORIDE BLEACHED, 1930 CROP,
KANRED FLOUR BAKED BY SPONGE AND DOUGH METHOD

Loaf number	Amount of	Date of baking					
	bleach	Aug. 21	Sept. 10	Sept. 19	Oct. 20	Jan. 24	
		Loaf volume	es in cubic co	entimeters			
1	Unbleached	1515	1510	1490	1490	1500	
	1/3 bleach	1515	1505	1480	1505	1520	
3	2/3 bleach	1587	1547	1515	1530	1555	
4	3/3 bleach	1555	1520	1520	1545	1550	
2 3 4 5	6/3 bleach	1567	1550	1535	1520	1535	
		Color	of crumb-sc	ore			
1	Unbleached	95	95	96	96	97	
2	1/3 bleach	96	98	96	97	97	
2 3	2/3 bleach	98	99	98	98	99	
4	3/3 bleach	98	99	98	99	99	
5	6/3 bleach	98	99	98	99	99	
		Text	ure of loaf-sc	ore			
1	Unbleached	93	97	96	97	98	
2	1/3 bleach	97	97	97	98	98	
2 3 4 5	2/3 bleach	98	98	97	98	98	
4	3/3 bleach	98	97	98	98	98	
5	6/3 bleach	98	. 98	98	98	98	

## Chlorine (Beta Chlora) Bleached Flour

The baking data for the 1929 Kanred chlorine bleached (Beta Chlora) flours are shown in Table V.

Only the full and double bleached flours were compared with the unbleached standard. In these tests a decrease in loaf volume was noted with the high rates of chlorine bleach, while the crumb color and texture seemed to remain fairly constant. The impaired loaf volumes were probably due to the fact that a shorter fermentation period should have prevailed. It is a well known fact that the higher the degree of treatment with either chlorine or Beta Chlora, the higher will be the hydrogen-ion concentration and the greater the need for a shorter fermentation period. A shorter fermentation period was not used in these tests, and a decrease in loaf volume probably was the result.

The baking results with the unbleached flour showed an improvement in crumb color and texture. The loaf volume, however, remained



Fig. 2. Loaves baked by sponge and dough method from nitrogen-trichloride bleached, Kanred wheat flour, crop 1930. Baked August 21, 1930. See Table IV for baking data.

TABLE V

RESULTS OF BAKING TESTS ON CHLORINE BLEACHED (BETA CHLORA), 1929 CROP, KANRED FLOUR BAKED BY THE SPONGE AND DOUGH METHOD

Loaf	Amount of		D	ate of bakin	ng	
number	bleach	June 21	June 28	July 11	July 25	Dec. 8
		Loaf volume	es in cubic ce	entimeters		
1	Unbleached	1563	1590	1612	1560	1575
2	3/3 bleach	1575	1598	1595	1512	1515
3	6/3 bleach	1575	1565	1552	1500	1525
		Color	of crumb-sc	ore		
1	Unbleached	94	94	96	97	97
2	3/3 bleach	97	96	97	98	98
2 3	6/3 bleach	98	98	98	99	99
		Textu	ire of Loaf-so	ore		
1	Unbleached	96	96	97	97	98
2 3	3/3 bleach	95	98	97	98	98
3	6/3 bleach			96	95	96

TABLE VI

RESULTS OF BAKING TESTS ON NITROGEN-TRICHLORIDE BLEACHED, 1930 CROP, KANRED FLOUR BAKED BY SPONGE AND DOUGH METHOD

Loaf	Amount of		Da	ate of baking		
number	bleach	Aug. 19	Sept. 10	Sept. 22	Oct. 22	Jan. 20
	1	Loaf volume	es in cubic ce	entimeters		
1	Unbleached	1507	1507	1510	1520	1540
2	1/3 bleach	1495	1505	1500	1525	1490
2 3	2/3 bleach	1483	1490	1463	1535	1510
4	3/3 bleach	1505	1520	1487	1540	1510
4 5	6/3 bleach	1455	1460	1440	1480	1500
		Color	of crumb-sc	ore		
1	Unbleached	95	96	97	97	97
2	1/3 bleach	97	97	98	98	98
2 3 4 5	2/3 bleach	98	97	98	98	98
4	3/3 bleach	99	99	99	99	99
5	6/3 bleach	99	97	99	99	99
		Textu	ire of loaf-sce	ore		
1	Unbleached	96	96	97	97	98
2	1/3 bleach	96	97	97	98	98
2 3 4 5	2/3 bleach	96	96	98	98	98
4	3/3 bleach	97	98	98	97	98
5	6/3 bleach	96	96	97	97	97

practically unchanged. A further aging of the flour due to storage did not improve the baking characteristics of the bleached flour.

In Table VI will be found the data on the chlorine bleached Kanred flour for 1930. As was the case with the 1929 Kanred flour bleached with chlorine there was a rather definite decrease in loaf volume in the double bleached flour from the very beginning of the investigation. This again may be partially explained as due to an increased H-ion concentration, with the fermentation period remaining unchanged. The crumb color and texture of the loaves did not decrease proportionally with the decrease in loaf volume. A maximum color of the crumb was obtained at the very beginning, and only a slightly inferior texture was noticeable in the double bleached flour as indicated by the score. Figure 3, illustrates the character of the loaves baked from the chlorine bleached flours on August 19.

The data obtained on January 26, 1931, compare favorably with those obtained on other days, namely, that no further development either better or inferior could be noted as due to a longer period of storage.

### RESULTS WITH STRAIGHT DOUGH METHOD

The baking results obtained with the various bleached flours by use of the two hour, short-fermentation, straight dough method, were similar in characteristics to those exhibited by the sponge and dough method. By this method a somewhat lesser loaf volume was obtained with all loaves. Aging in storage was responsible for definite increases in loaf volume, crumb, and texture scores, with the possible exception of the highly bleached (chlorine) flours which showed properties similar to those baked by the sponge and dough method. Excessive applications of nitrogen-trichloride and benzoyl peroxide did not seem to impair the baking quality of the flour.

# RESULTS BY THE POTASSIUM BROMATE DIFFERENTIAL METHOD

Nothing significant with respect to the action of bleaching agents on flour developed by the use of the potassium bromate differential method, as can be seen by consulting Table VII. There was very little variation on loaf volume, crumb texture, and color, due to the presence of potassium bromate with any of the Novadel bleached flours. This would further prove that benzoyl peroxide is not a harmful oxidizing agent, even when used in excessive amounts. Photographs illustrating the baking results are shown as Figure 4.

# Nitrogen-Trichloride Bleached Flours

The baking results obtained by use of the potassium bromate differential method with Kanred flours bleached with nitrogen-

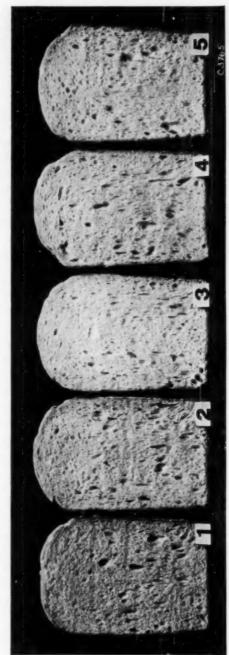


Fig. 3. Loaves baked by sponge and dough method from chlorine (Beta Chlora) bleached, Kanred wheat flour, crop 1930. Fig. 3.

TABLE VII

RESULTS OF BAKING TESTS ON BENZOYL PEROXIDE BLEACHED, 1930 CROP, KANRED FLOURS BAKED BY THE POTASSIUM BROMATE DIFFERENTIAL METHOD

Amount of bleach	Kind of bleach	KBrO <sub>3</sub> added	Year	Loaf volume	Color score	Texture	
				cc.			
3/3	Benzoyl-peroxide	None	1929	1565	99	97	
3/3	Benzoyl-peroxide	1 mg.	1929	1530	99	97	
3/3	Benzoyl-peroxide	2 mg.	1929	1580	99	98	
6/3	Benzoyl-peroxide	None	1929	1540	99	98	
6/3	Benzovl-peroxide	1 mg.	1929	1560	99	99	
6/3	Benzoyl-peroxide	2 mg.	1929	1590	99	98	
3/3	Benzoyl-peroxide	None	1930	1570	99	95	
3/3	Benzoyl-peroxide	1 mg.	1930	1555	99	95	
3/3	Benzoyl-peroxide	2 mg.	1930	1530	98	97	
6/3	Benzoyl-peroxide	None	1930	1530	99	95	
6/3	Benzoyl-peroxide	1 mg.	1930	1530	99	97	
6/3	Benzoyl-peroxide	2 mg.	1930	1540	98	95	

trichloride show clearly that the baking quality of the flour was impaired when potassium bromate was added to the flours which had a full and double bleach by this reagent. Not only was there a pronounced decrease in loaf volume but there was a similar decrease in texture and color of crumb. These findings are recorded in Table VIII and illustrated in Figure 5.

TABLE VIII

RESULTS OF BAKING TESTS ON 1929 AND 1930 CROP KANRED FLOURS BLEACHED WITH NITROGEN-TRICHLORIDE AND BAKED BY THE POTASSIUM BROMATE DIFFERENTIAL METHOD

Amount of bleach	Kind of bleach	KBrO₃ added	Year	Loaf volume	Texture score	Color
				cc.		
3/3	Nitrogen-trichloride	None	1929	1550	99	98
3/3	Nitrogen-trichloride	1 mg.	1929	1500	99	98
3/3	Nitrogen-trichloride	2 mg.	1929	1470	98	98
6/3	Nitrogen-trichloride	None	1929	1540	99	99
6/3	Nitrogen-trichloride	1 mg.	1929	1535	99	99
6/3	Nitrogen-trichloride	2 mg.	1929	1240	92	95
3/3	Nitrogen-trichloride	None	1930	1480	98	99
3/3	Nitrogen-trichloride	1 mg.	1930	1505	98	98
3/3	Nitrogen-trichloride	2 mg.	1930	1420	96	98
6/3	Nitrogen-trichloride	None	1930	1515	98	99
6/3	Nitrogen-trichloride	1 mg.	1930	1440	98	99
6/3	Nitrogen-trichloride	2 mg.	1930	1370	92	95

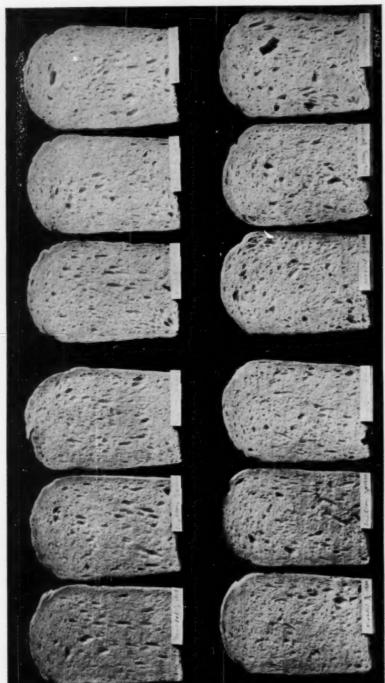


Fig. 4. Loaves baked by potassium bromate differential method on 1929 and 1930 crop Kanred flours bleached with varying amounts of benzoyl peroxide. amounts of peroxide used as well as the milligrams of bromate added to the doughs are shown on the plate. Baking data are recorded in Table VII.

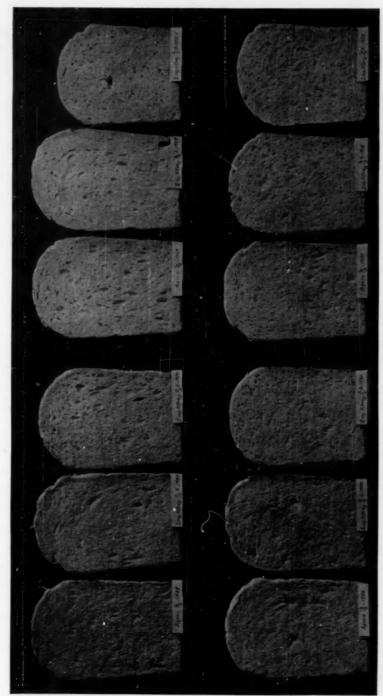


Fig. 5. Loaves baked by the potassium bromate differential method on 1929 and 1930 Kanred flours bleached with varying amounts of nitrogen-trichloride. The extent of the bleach as well as the milligrams of potassium bromate added to the dough are shown on the plate. Baking data are recorded in Table VIII.

### Chlorine (Beta Chlora) Bleached Flours

The chlorine bleached Kanred flour of 1930 baked January 31, 1931 by the potassium bromate differential baking test shows a decided decrease in loaf volume, as well as texture of crumb. The crumb color does not seem to decrease proportionally with the loaf volume, as indicated by the score.

The fact that chlorine is such an active bleaching reagent accounts for the low volume of the loaves due to oxidation.

The unbleached Kanred flour of 1929 and 1930 baked by the same method shows a distinct improvement in loaf volume, color of crumb, and texture by successive additions of one and two milligrams of potassium bromate. The baking data are shown in Table IX and photographs of the loaves are shown in Figure 6.

TABLE IX RESULTS OF BAKING TESTS WITH 1929 AND 1930 CROP KANRED FLOURS BLEACHED

WITH CHLORINE (BETA CHLORA) AND BAKED BY THE POTASSIUM BROMATE DIFFERENTIAL METHOD Amount of Kind of KBrO. Loaf Color

bleach	bleach	added	Year	volume	score	score	
				cc.			
3/3	Chlorine	None	1930	1520	99	98	
3/3	Chlorine	1 mg.	1930	1500	99	98	
3/3	Chlorine	2 mg.	1930	1460	99	94	
6/3	Chlorine	None	1930	1385	99	94	
6/3	Chlorine	1 mg.	1930	1370	99	90	
6/3	Chlorine	2 mg.	1930	1300	99	90	
None	Unbleached	None	1929	1300	95	88	
None	Unbleached	1 mg.	1929	1400	95	92	
None	Unbleached	2 mg.	1929	1445	97	98	
None	Unbleached	None	1930	1490	95	96	
None	Unbleached	1 mg.	1930	1590	97	98	
None	Unbleached	2 mg.	1930	1520	97	98	

# THE RESULTS WITH MECHANICAL METHOD OF MODIFYING DOUGH

The results obtained from the baking of the bleached Kanred and Blackhull flours by the methods just described clearly showed that no appreciable difference existed between the two flours as the result of bleaching. The mechanical treatment showed a greater effect from variety than from bleaching. Blackhull is particularly sensitive to excessive mechanical treatment.

### RESULTS OF LABORATORY TESTS

Viscosity readings in degrees (MacMichael) showed a fairly uniform and consistent average with all flours. No appreciable differences

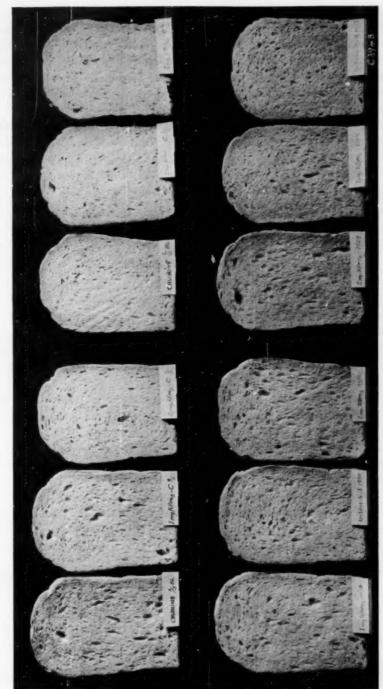


Fig. 6. Loaves baked by the potassium bromate differential method on 1929 and 1930 Kanred flours bleached with varying amounts of chlorine (Beta Chlora). The extent of the bleach as well as the milligrams of potassium bromate added to the dough are shown on the plate. Baking data are recorded in Table IX.

could be detected except for the flours having high rates of chlorine bleach. These gave a low viscosity reading. No definite correlation existed between the viscosity readings and the loaf volume, although a lower reading of the full and double bleached chlorine flours compared favorably with a lower loaf volume of these bleaches. The viscosity readings of the unbleached standard seemed to increase slightly with the age of the flour.

The acidity of the flours did not show any appreciable change when bleached with the Novadel and Agene. A slight increase in hydrogenion concentration, however, was noted in the chlorine bleached flours. The hydrogenion determination of April 15, 1931 showed a slight increase in acidity of all the bleached flours due to storage.

Note. After the work was started, results showed that the flours from these two crops were not as sensitive to bleaching agents as the flours from certain other crops. This means that effects of bleaching were less than would have been with flours from some other crops.

# Summary and Conclusions

1. The yellow color of flour, due to carotin, is slowly destroyed by oxidation when flour is stored for a considerable time. It is also destroyed by chemical substances used in the commercial bleaching of flour of which the most common are: Nitrogen peroxide (Alsop), nitrogen trichloride (Agene), chlorine (Beta Chlora), and benzoyl peroxide (Novadel). These substances also produce a maturing effect similar to natural aging.

2. Flours were milled from wheats of the 1929 and 1930 crops so as to compare the effect of the aging of the wheat with the aging of flour. These flours were bleached with Agene, Beta Chlora, and Novadel using the following rates:  $0, \frac{1}{3}, \frac{2}{3}, \frac{3}{3}$ , and 6/3 based on the amounts recommended for commercial practices. The flours were then stored in a well ventilated room.

3. These flours were baked from time to time using several methods. At first the intervals between baking were about a week, then longer, and toward the last, several months. The total time of storage of the several flours was between six and nine months. Viscosity and hydrogen ion determinations were also made.

4. No detrimental effects on the baking results were noticeable with any of the bleaches used with the possible exception of chlorine; and detrimental effects on baking quality were noted only when high rates of bleach were used. Viscosity was slightly lowered only when the full and double chlorine bleaches were used. The hydrogen ion concentration was also increased with this bleach.

5. The volume, texture, and crumb color in the bread made from the flours milled from the wheat one year old were somewhat better than those from the flours milled from the wheat recently harvested. This indicates a beneficial aging effect in the wheat.

6. Nitrogen-trichloride and chlorine so condition the flour that optimum baking results are obtained on flour from new wheat. Unbleached flour showed a noticeable improvement in baking qualities from aging. This was not apparent in the bleached flours. The addition of potassium bromate to the fully bleached and double bleached flour did not show any beneficial results and in some cases it produced harmful effects.

Bibliography of Bibliographies on Chemistry and Chemical Technology, Second Supplement, 1929–1931. Bulletin 86, The National Research Council, Washington, D. C., March, 1932. 150 pp. Compiled by Clarence J. West and D. D. Berolzheimer. Price \$1.50.

This bulletin is the second supplement to Bulletin 50 of the same title, which was published in 1925. Bulletin 71, also of the same title, was published in 1929. The bulletin lists bibliographies on some 1075 special subjects scattered throughout the sciences. There is, likewise, reference to 60 personal bibliographies, as well as a reference to a general work regarding 68 chemists. To be most helpful, users in search of information regarding published bibliographies should consult the three bulletins comprising the series, i.e., Bulletins 50, 71, and 86. To be of value to the cereal chemist, there is much need for strengthening the subject matter in this field of chemistry. A number of useful bibliographies on milling and baking as well as outstanding original researches, bibliographical in type and which were published during the period of compilation, have been overlooked.

However, as a reference work to other lines of research, the bulletin will be found useful.

D. A. COLEMAN.

# SUGARS, DIASTATIC ACTIVITY, AND "GASSING POWER" IN FLOUR 1

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(Read at the Convention, May, 1932)

That flours vary widely in their ability to maintain gas production during yeast fermentation under conditions where all other factors are equal is a fact now thoroughly established and generally recognized. This inherent flour property is commonly designated by European workers as gassing power, and, as would be expected, it is positively correlated with diastatic activity. The importance of giving due recognition to this factor in laboratory test baking has recently been emphasized by various workers, and for the purposes of this communication no further reiteration of this point is necessary.

It is customary to regard the terms gassing power and diastatic activity as synonymous. Thus, for example, Jørgensen (1931) expresses the opinion that the term 'gassing power' may be substituted by the term 'diastatic activity.' Strictly speaking, however, gassing power is more inclusive than diastatic activity. The latter refers exclusively to the flour's ability to convert starch into maltose, saccharogenesis, being the term appropriately used by Bailey (1925). The former includes not only saccharogenesis, but also the fermentable sugars originally present in the flour. To use the two terms interchangeably is necessarily to assume that all flours have essentially the same original sugar content, and that differences in gassing power are due solely to different diastatic or saccharogenic powers. Whether or not this is actually and generally true has never been established, to the writers' knowledge.

Several considerations have suggested the studies that are to be presented and discussed in this report. 1. The senior author is serving as Associate Referee on Diastatic Activity of Flour for the Association of Official Agricultural Chemists, and from that standpoint there are several matters that need clearing up in the interests of the ultimate establishment of a suitable standard procedure for the quantitative estimation of diastatic activity. 2. Occasional (though infrequent) exceptions have been noted to the general situation re-

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ported by Blish and Sandstedt (1927), and by Blish, Sandstedt, and Platenius (1929), who found a decided positive correlation between crust color and diastatic activity, when loaves were baked according to the American Association of Cereal Chemists basic procedure. Some instances have been encountered in which there was slightly more or less crust color than the actual diastatic values seemed to warrant. Were these instances due to variations in original sugar content of the flour?

## Sugars in Flour

The estimation and identification of sugars in biological materials have always been attended with difficulties and uncertainties. This is evidenced by the large constantly increasing number and variety of analytical procedures and modifications of procedure for the estimation of sugar in both plant and animal products that are to be found in current as well as in the back files of all technical journals dealing with the biochemical subjects. A critical discussion of various types of methods and of complications involved is outside the scope of this paper.

For present purposes it is enough to say that in the estimation of sugars in biological material the results usually vary not only with the method used, but also with the manner in which the material is prepared for analysis. Wheat flour is no exception to this rule, as judged from the limited amount of data that writers have been able to find in the literature available to them.

There is in the latest A. O. A. C. Book of Methods no official or tentative procedure for the estimation of sugars in cereal foods. The preparation of the flour extract specified in the A. A. C. C. Book of Methods is essentially the same as the A. O. A. C. method for use with cattle feeds. It involves clarification with lead acetate after preliminary extraction with alcohol and does not work satisfactorily with wheat flour, as was also noted by Rumsey (1922).

Rumsey's (1922) detailed literature review refers to a number of workers who have had occasion to study the carbohydrate content of wheat flours. These workers conclude in general that the amount of soluble sugars normally present as such in flour is insignificant.

As a matter of fact, the existing published data regarding sugar content of flour are somewhat contradictory. Among the earlier American data are those of Stone (1896). He examined both spring and winter wheat flours, and reported no reducing sugars, with not to exceed 0.2% sucrose. Teller (1912) reported figures of similar magnitude. These are the lowest values that the writers have encountered in the literature. Stone and Teller extracted their flours with hot 95%

alcohol, evaporated off most of the alcohol, took up the residue with water and applied the Fehling's solution type of method.

Shutt (1908) found considerably larger quantities of soluble sugar in flour. He extracted for 12 hours with 70% (by weight) alcohol, and applied a Fehling solution procedure to the extracts. He reports from 0.05% to 0.26% of reducing sugar, as maltose, and from 0.91% to 1.42% sucrose. He found much greater quantities by extracting with water, but the increase must have been due to diastasis during extraction.

Rumsey (1922) reported values for reducing sugars in 14 different flour samples. When the values in his Table XVIII are calculated to per cent of maltose they show a range of about 0.2% to 0.7%, the average being approximately 0.4%. He extracted the flour samples with water containing sodium tungstate, the latter being added for the intended purpose of rendering the diastases inactive during the period of extraction.

Rumsey's values for reducing sugars in flour are larger than any of the others mentioned. This arouses a suspicion that his specified treatment with sodium tungstate does not entirely inhibit diastatic action and that his higher values for reducing sugars are due, in part, to a slight diastasis during extraction. That this is indeed the case was

later reported by Malloch (1929).

In spite of the lack of agreement among existing data with respect to the reducing sugar content of flours, it is apparent that in normal sound flours the quantity is very small. Nevertheless, an accurate knowledge of the true situation is essential from the standpoint of diastatic activity determinations by the Rumsey (1922) procedure or any of its modifications, in view of the general and widespread adoption of this type of method for use in diastatic activity investigations in recent years.

Rumsey's (1922) type of method involves two main steps. The first is the *blank* determination, in which the reducing sugar (calculated as maltose) originally present in the flour, is estimated in a water extract obtained under conditions in which the diastase has been purposely rendered inactive. The second step is a similar estimation of maltose in the extract from a flour-in-water suspension in which diastatic action has been allowed to proceed for one hour. The difference between the two values represents the maltose produced by diastatic action.

In the Rumsey type of procedure it is obviously a matter of first importance to accurately and reliably establish the true value for the blank, representing the reducing sugars originally present in the flour. The facts, as previously mentioned, that various workers have not been in satisfactory agreement as to the quantity of reducing sugars normally and actually present as such in bakers' flours, and that the values reported vary according to the method employed for their extraction, would seem to warrant further inquiry into the situation.

Existing data, which have been confirmed and verified by our own experience, show that if extraction with boiling 95% alcohol is a reliable procedure, then there is certainly not to exceed the merest trace of reducing sugar originally present as such in sound, normal bakers' flour. The quantity extracted by this method is so minute that it may be utterly disregarded. When dilute alcohol (70%) is used for flour extraction, very small but, nevertheless, measurable amounts of reducing sugars are found. Methods involving extraction with water have ordinarily yielded appreciably higher results in spite of precautions devised for the purpose of preventing any diastasis during the extraction.

Precautions used for preventing diastatic activity during water extraction have included the following: (1) Addition of acid or alkali for the purpose of producing a hydrogen ion concentration unfavorable to diastatic action; (2) extraction at low temperatures; (3) addition of salts of heavy metals, such as sodium tungstate; and (4) a combination of two or more of these treatments. In the light of modern knowledge of sugar chemistry and of enzyme behavior, however, few of these methods are entirely exempt from all possibility of criticism. Extraction with ice-water alone apparently does not completely stop all diastatic action, and the same holds true for the use of sodium tungstate. A concentration of acid that is sufficient to completely stop all possibility of diastatic action is likely to cause some inversion of sucrose. Alkali, on the other hand, is known to decompose sugars under certain conditions, its action being more rapidly destructive with the aldehyde sugars than with sucrose.

It is the belief of the writers that a suitable procedure involving extraction with water is preferable to alcohol extraction for several reasons, that for present purposes need no discussion. The first step in this series of studies has, therefore, been an attempt to find a convenient water extraction procedure under conditions in which there is satisfactory elimination, not only of diastasis during extraction, but also of any appreciable hydrolysis or decomposition of sugars present.

# Experimental

As Associate Referee on the Diastatic Value of Flour for the A. O. A. C., the senior author (Blish, 1931) last year conducted a series of studies involving a modified Rumsey (1922) procedure, working toward the establishment of a basis for the later adoption of an official method.

The first consideration was the inactivation of the flour diastase in the blank determination which involves the estimation of reducing sugar originally present as such in the flour.

Due recognition was taken of Malloch's (1929) observation that the presence of sodium tungstate, alone, during extraction for the blank determination, does not completely stop diastasis, and that sulfuric acid must be used in conjunction with the tungstate. A series of 26 miscellaneous baker's flours was tested, and the procedure was to extract 10 gms. of flour with 100 cc. of water containing 3 cc. of concentrated H<sub>2</sub>SO<sub>4</sub> and 5 gms. of sodium tungstate per liter for 1 hour at room temperature, shaking every 15 minutes.

Using a picric acid colorimetric procedure for the estimation of reducing sugars, calculated to maltose, with the exception of one flour which gave a value of 0.39% maltose, all values fell between 0.30% and 0.36%, the average value being 0.33%. Closely agreeing values were secured when the copper reduction method was substituted for the colorimetric procedure, the latter being preferred as more convenient and time-saving. These values fall in between the average of values obtained by Rumsey (1922) and those of Malloch (1929), although the latter reported data on only one flour. They are far more consistent and uniform, however, than Rumsey's data on 14 flours, in which values ranged from 0.2% to 0.7%, as previously noted. They indicate that baker's flours are very uniform as to the reducing sugar content.

The results of these studies were reported at the 1931 meeting of the A. O. A C. More recently, these studies have been extended and have involved additional modifications of treatment. The further and more recent studies show that in all probability the values of 0.3% to 0.4% reducing sugars are themselves too high, the actual quantity being, on the average, a little more than one-half that amount.

It has proven difficult to establish ideal conditions for the extraction of reducing sugars with water. When the extraction is conducted at room temperature, and sufficient acid is added to completely inhibit all diastatic activity, there is a likelihood of some hydrolysis of sucrose by the acid. Conversely, if acid concentration is kept low enough to avoid any sucrose inversion, diastasis may not be completely inhibited. Extraction with ice-water alone does not completely stop diastatic activity. Optimum results were obtained only by a combination of ice-water temperature and a certain degree of acidity, and conditions were finally established under which both diastatic activity and hydrolysis of sucrose are probably at an almost irreducible minimum

in so far as water extraction is concerned. A few typical experimental data are here presented.

Table I, shows the effect of varying the concentration of H<sub>2</sub>SO<sub>4</sub> on values in the Rumsey blank determination. Each extract contained 5 gms. of sodium tungstate per liter, and all extracts were filtered and subjected to maltose determination immediately after the expiration of the 1 hour digestion period. The digestions were at room temperature.

TABLE I

EFFECT OF VARYING H<sub>2</sub>SO<sub>4</sub> Concentration on Maltose in Rumsey Blank
Determination

Concentrated H <sub>2</sub> SO <sub>4</sub> per liter (cc.)	1	3	5	7	9
Maltose per 10 gms. of flour (mgs.)	43.1	27.9	35.5	41.0	50.9
pH of extracts	2.9	1.4	-	-	-

These data indicate that 1 cc. of H<sub>2</sub>SO<sub>4</sub> per liter of extracting solution containing the specified quantity of sodium tungstate was not sufficient to completely inhibit diastatic activity. The pH was 2.9, while Rumsey's (1922) data show that a pH of below 2.0 is necessary for the complete inhibition of diastasis at 27° C. The lowest maltose figure was obtained with 3 cc. of H<sub>2</sub>SO<sub>4</sub> per liter, the values again increasing progressively with the higher concentrations of H<sub>2</sub>SO<sub>4</sub>. This suggests that sucrose hydrolysis occured with concentrations of 5, 7 and 9 cc. of H<sub>2</sub>SO<sub>4</sub> per liter, and that the value obtained with the concentration of 3 cc. per liter is more nearly the correct one. There is no assurance, however, that no sucrose inversion occurred even in this instance.

Another series was run in which 2 to 3 hours time elapsed between the filtration of the extract and the withdrawal of aliquots for sugar determinations. Here the maltose figures were appreciably higher than in Table I, indicating that further hydrolysis of sucrose had occurred.

Further experiments showed that minimal values for reducing sugars in water extracts of flour could be secured only by combining the use of acid and ice-water temperatures during extraction.

Table II shows results of a typical series of experiments involving the use of different acid concentrations at ice-water temperatures.

TABLE II

EFFECT OF VARYING H<sub>2</sub>SO<sub>4</sub> Concentrations on Maltose in Ice-Water Extractions

Concentrated H <sub>2</sub> SO <sub>4</sub> per liter (cc.)	1	2	3	4	5
1 hour extraction— Maltose per 10 gms. flour (mgs.)	21.5	18.9	16.9	15.6	15.2
2 hour extraction— Maltose per 10 gms. flour (mgs.)	20.6	18.3	17.0	16.8	

The data in Table II show no evidence that there could have been either diastasis or sucrose inversion under these conditions, corresponding values being the same for the 2-hour as for the 1-hour extraction. The slight reduction in values with increasing acid concentration cannot, at present, be explained by the writers, nor is there any assurance as to which value, if any, is the correct one.

It is possible that the different acid concentrations influenced the picric acid colorimetric estimations of maltose in some manner which the writers have not yet been able to ascertain. The values are so small that the colorimetric procedure is preferable to the copper reduction method.

Under any circumstances it appears reasonable to interpret these data as showing that the actual reducing sugar (calculated as maltose) content of the flour under consideration is not more than 0.15% to 0.21%.

The next step was to determine reducing sugars in a series of 33 baker's flours using H<sub>2</sub>SO<sub>4</sub> at ice-water temperatures. The series included representative, samples milled commercially from Northwestern, Southwestern, Pacific Coast and Canadian wheats. The procedure was to add to 5 gms. of flour in an Erlenmeyer flask 25 cc. of water containing 6 cc. of concentrated H<sub>2</sub>SO<sub>4</sub> per liter, followed by 25 cc. of 1% sodium tungstate, giving a final concentration of 3 cc. H<sub>2</sub>SO<sub>4</sub> and 5 gms. sodium tungstate per liter. The flask and ingredients were individually cooled to ice-water temperature before mixing. Digestion was performed in an ice-water bath, for 1 hour, with occasional shaking. The extracts were then filtered and aliquots were at once taken for maltose estimations by the picric acid colorimetric method. The data obtained by using this procedure on the 33 flours are shown in Table III.

TABLE III
REDUCING SUGARS ORIGINALLY PRESENT IN 33 REPRESENTATIVE BAKER'S FLOURS

Flour number	Maltose	Flour number	Maltose	Flour number	Maltose
	P.ct.		P.ct.		P.ct.
2	0.15	15	0.11	26	0.17
5	.15	16	.15	27	.15
6	.13	17	.15	28	.15
7	.13	18	.15	29	.04
8	.13	19	.14	30	.14
9	.12	20	.18	31	.12
10	.12	21	.18	32	.13
11	.14	22	.16	33	.14
12	.13	23	.19	34	.13
13	.16	24	.19	35	.16
14	.10	25	.14	36	.14

The data in Table III strongly support the viewpoint that the content of reducing sugars in flours is actually very small, and nearly constant within the limits of experimental error of quantitative estimation. If the situation is as here represented, then the usual blank determination in estimating diastatic activity by the Rumsey (1922) method is, for all ordinary purposes, an unnecessary step. It is suggested that the blank determination be dispensed with entirely, and that a constant value of, say, 15 mgs. of maltose per 10 gms. of flour, be arbitrarily subtracted from the total maltose present after 1 hour's diastasis.

This should eliminate about one-half of the operations of the Rumsey method without significantly detracting from reliability of results. Only in special instances, as, for example, where the flour is suspected of being unsound, or milled from frosted or otherwise damaged wheat, should the Rumsey blank determination be necessary.

### Sucrose Content of Flours

As has been previously noted, the fact that individual flours may show a certain order of variation in diastatic power, does not necessarily insure that their comparative gassing powers will rate in the same order. The order of rating could be the same for both properties only in the event the flours all had the same original sugar content, since a flour's gas producing capacity involves not only fermentable sugars produced by diastasis but also the total quantity of fermentable sugar already present in the flour. It has been shown to be extremely probable that the great majority of baker's flours, if milled from sound wheat, have a nearly constant and almost negligible content of reducing sugars. Does a similar situation apply to the sucrose content?

The foregoing question was suggested by observations of test loaves from a group of flours baked without the use of sugar in the formula, occasional instances being noted where comparative gassing powers and crust colors deviated slightly from the order that was indicated by individual diastatic value determinations. Accordingly, it was deemed advisable to determine the sucrose content in the series of flours listed in Table III.

For this purpose the flours were extracted in the manner suggested as most suitable for the estimation of reducing sugars in the blank determination for diastatic activity. Fifty cubic centimeters of extract were treated with 5 cc. of HCl for inversion and allowed to stand overnight at room temperature. Total invert sugar was then estimated by the Munson and Walker copper method, and the increase above the amount of reducing sugar found in the blank (calculated as

invert sugar) was calculated to sucrose. These sucrose values are reported in Table IV, which follows:

TABLE IV Sucrose Content of Flour Series

Flour number	Sucrose	Flour number	Sucrose	Flour number	Sucrose
	P.ct.		P.ct.		P.ct.
2	1.01	15	1.34	26	1.10
5	1.17	16	1.15	27	1.07
6	1.44	17	1.74	28	1.25
7	1.56	18	1.00	29	1.22
8	1.15	19	1.34	30	1.17
9	1.53	20	1.34	31	1.23
10	1.49	21	1.11	32	1.40
11	1.10	22	1.28	33	1.18
12	1.00	23	1.15	34	1.19
13	1.16	24	1.27	35	1.28
14	1.38	25	1.28	36	1.30

The values in Table IV are of the same order of magnitude as sucrose figures reported by Shutt (1908). From these data, which involve 33 representative baker's flours from various parts of the United States and Canada, it is evident that sucrose is ordinarily present in quantities large enough to significantly influence the gassing power of flour. The sucrose values also show a fairly wide range of variation, the range in this series being from 1% to 1.74% of sucrose. This means that on a basis of equal diastatic activities, flour No. 17, with 1.74% of sucrose should have a significantly higher gassing power than flour No. 18, which sucrose content was 1%.

# Diastatic Activity

A modified Rumsey (1922) procedure for diastatic activity was applied to the series of 33 flours. This involved estimations of total maltose present after diastasis for 1 hour, under the following conditions: 5 gms. flour, 47.8 cc. of buffer solution,<sup>2</sup> and a teaspoonfull of ignited quartz sand (to facilitate breaking up lumps of flour without excessively violent shaking) were introduced into a 100 cc. Erlenmeyer flask, all materials having previously been brought to 30° C. Digestion was allowed to proceed for 1 hour at 30° C. in a water thermostat, the contents of the flask being mixed by rotating the flask a few times every 15 minutes. At the end of the hour, 0.2 cc. of concentrated H<sub>2</sub>SO<sub>4</sub> were added, followed by 2 cc. of a 12% sodium tungstate solution, with thorough mixing after each addition. As soon as possible the extract was filtered, discarding the first few 2 or 3 cc. The extract was

<sup>&</sup>lt;sup>2</sup> Suggested by Malloch (1929). Mix 8 parts of a solution containing 21.008 gms. of crystalline citric acid and 200 cc. of N/1 NaOH per liter with 2 parts of 0.1N HCl. Dilute 1 part mixture with 3 parts of water. This gives a pH of about 4.7.

immediately subjected to maltose estimation by a picric acid colorimetric procedure as described in a recent report to the A. O. A. C. by Blish (1931).

Values for total maltose found in the flour extracts by the foregoing procedure, for 33 flours, are shown by the *broken* line in Figure 1. Each point on the horizontal axis represents an individual flour, while the values along the vertical axis indicate mgs. maltose per gms. of flour.

The continuous line in Figure 1 shows diastatic values obtained by actually subtracting the blank figures (shown in Table III, and indicating maltose originally present in the flours, respectively) from the corresponding values for total maltose after diastasis for 1 hour (shown by the broken line in Figure 1) as called for in the Rumsey

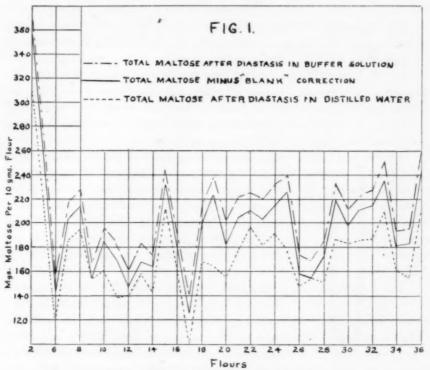


Fig. 1. Maltose values by three different methods.

(1922) method. The two graphs parallel each other closely at all points. This strongly substantiates the conviction, previously stated, that for estimating comparative diastatic activities by the Rumsey (1922) type of procedure, the so called blank value may be considered as a constant, and its separate estimation is an unnecessary step.

The dotted line in Figure 1 also shows total maltose values after diastasis for 1 hour, but in this series of determinations the liquid medium was distilled water alone, instead of the buffer solution. This gives somewhat lower values than were obtained with the buffer solution pH 4.7, but the order of differences among the individual flours is substantially the same as in the series with the buffer solution. This suggests that hydrogen ion concentration is not ordinarily a factor of serious consequence in the estimation of comparative diastatic powers of flours. Nevertheless, the elimination of all possible variables other than the one under consideration is always a desirable feature, and for this reason, together with the fact that higher values are obtained with the buffer solution, the use of the buffer solution is here given preference.

### Gassing Power

The types of methods most commonly used for testing both quantity and rate of gas production have been recently referred to by Jørgensen (1931). Perhaps the most familiar type of procedure is that of Jago (1911), in which the dough or flour-yeast-water suspension is allowed to ferment in a bottle or flask, the CO<sub>2</sub> produced forces liquid from a second flask or bottle over into a graduated cylinder. As ordinarily used, this procedure is of somewhat doubtful precision, and yeast variability is also a factor.

Schultz and Landis (1932) recently reported on the use of a method for diastatic power in vegetable products in which yeast is added to the reacting solutions in sufficient quantity to ferment out the sugars as rapidly as they are formed and the carbon dioxide evolved is collected and measured at frequent intervals. Their method involved continuous shaking of the reacting mixture in a thermostat at 30° C. the gas volumes were measured in a suitable gasometer equipped with leveling bulbs.

Based somewhat upon the principle applied by Schultz and Landis (1932) a simplified precision method has been worked out for the estimation of the comparative gassing powers of flours. The method consists simply of treating a small quantity of flour with a relatively enormous excess of yeast, in water suspension, in a small flask fitted with a mercury manometer, the entire flask being immersed in a water thermostat at 30° C. The excess of yeast is so large that the sugars originally present are almost completely fermented out in 5 to 10 minutes, while the maltose developed by diastasis is fermented out practically as fast as it is formed. At the end of a suitable period, say 1 hour, the flask is vigorously shaken, and the pressure caused by the carbon dioxide developed is read on the manometer and recorded in

millimeters. The pressure developed is directly proportional to the gassing power of the flour.

As used at the present time, the details of the procedure are briefly as follows: 3 gms. of flour and approximately 10 gms. of quartz sand are placed in a 125 cc. Erlenmeyer flask and mixed. Thirty-five cubic centimeters of water are added and the flask is gently shaken until all the flour is thoroughly in suspension, and 6 drops of oleic acid <sup>3</sup> are introduced. Twenty-five cubic centimeters of a smooth suspension of bakers' yeast in water (prepared previously with a malted milk stirrer), containing 60 gms. yeast to 100 cc. of water are then withdrawn by means of a pipette (with constant agitation of the yeast suspension during withdrawal) and added to the flour suspension in the

flask. The flask is at once shaken for not more than 4 or 5 seconds, and the manometer, which is merely an S-shaped glass tube containing mercury and fitted into a rubber stopper, is at once inserted into the neck of the Erlenmeyer The rubber stopper is first moistened with water and must then be forced into the flask as tightly as is possible. The forcing of the stopper into the flask causes several centimeters of pressure to register in the manometer. The upper mercury level is immediately marked with a glass marking pencil or a small piece of gummed paper, and the flask is allowed to remain immersed, by means of lead weights, in the water thermostat for 1 hour. At the end of the hour the flask is vigorously shaken until the top mercury level in the manometer remains constant. Usually not more than 15 to 20 seconds of vigorous shaking are necessary. The rise of the mercury level above the original mark is measured with a millimeter rule, and this value doubled gives the millimeters of pressure produced.

Figure 2 shows the flask fitted with the manometer. The manometer is of thick walled glass barometer tubing with a 4 mm. bore. The flask and all ingredients are individually brough

FIG.2.

Fig. 2. Apparatus for determining comparative "gassing powers" of flours.

flask and all ingredients are individually brought to the temperature of the thermostat before the determination is initiated. It is, of course, essential that the volume of the flask, as well as its contents, be kept

<sup>&</sup>lt;sup>2</sup> This prevents foaming during the final shaking at the close of the digestion period. Caprylic alcohol, amyl alcohol, and related substances are not suitable as they apparently undergo fermentation, and cause greatly increased pressure.

constant at all times so as to afford comparative conditions for gas expansion.

The foregoing method has been found to give very satisfactory results, although there are possibilities for further improvements which should make for greater convenience, if not for higher precision. The variability of the method was tested by running a series of 12 determinations on flour 2, which had by far the highest diastatic power of the series of flours involved in these studies.<sup>4</sup> The data are shown in Table V.

TABLE V
VARIABILITY OF "GASSING POWER" METHOD

Determination number	Pressure mm.	Determination number	Pressure mm.	
1	206	7	205	
2	210	8	197	
3	213	9	210	
4	204	10	204	
5	208	11	198	
6	210	12	204	
Mean	*		206	
Standard deviation	n		$\pm 4.65$	
P.E. of single dete			$\pm 3.14$	
Coefficient of varia	ation	3	2.25	

The comparative gassing powers of the 33 flours, respectively, as estimated by this procedure, are given in Figure 3, each point on the continuous line graph representing an average of two closely agreeing determinations. The diastatic activities are again graphically shown by the broken line, in order to furnish a convenient basis for comparing the two sets of values. It should be noted that the values along the vertical axis serve a dual purpose. They represent milligrams of maltose as applied to the diastatic activity graph, and millimeters of mercury as applied to the gassing power graph.

Inspection of Figure 3 shows that the two graphs have many characteristics in common. For the most part, there is a close parallelism between diastatic activity and gassing power. There are certain deviations, however, that can be readily explained by variations in original sucrose content of the flours in question. Thus, for example, flour 17 is lowest of all in diastatic activity, but it happens to run highest of all in sucrose content (see Table IV) and is, therefore,

<sup>&</sup>lt;sup>4</sup> During the experiments here reported, values obtained with a given flour were essentially the same on different days and with different batches of yeast. Subsequent to the completion of these experiments, and since this paper went to press, some variability among different lots of bakers' yeast has been encountered, some lots giving substantially and consistently lower "gassing power" values despite the relatively enormous excess of yeast employed. Studies are now in progress in an effort to identify and eliminate the cause of this occasional deviation from normal values. Until this factor can be eliminated completely, however, it is advisable to make a preliminary test of each new lot of yeast with a standard flour, and introduce a correction factor if necessary.

only 14th from the lowest in gassing power. A similar situation applies to flour 6. It is second lowest in diastatic power, but ranks much higher in gassing power, due to high sucrose content, and there are no less than 12 flours below it in gassing power. Thus it becomes evident that, in a strict sense, the terms diastatic activity and gassing power are not synonymous as is commonly supposed.

There is justification for believing that a gassing power determination by the method here suggested will present a better picture of a

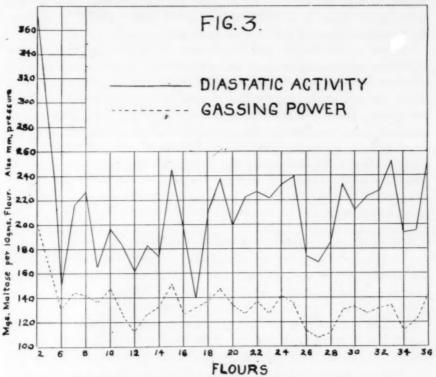


Fig. 3. Comparative "gassing power" and "diastatic activity" values of 33 baker's flours.

flour's inherent potentiality for supporting yeast fermentation (fermentation tolerance) than will a diastatic activity determination by the Rumsey (1922) type of method. The simplicity and compactness of the apparatus makes for precision in the control of temperature, and materially increases the number of determinations that can be made in one day.

Then there is the possibility for using the procedure to indicate comparative diastatic values alone. If the flask is shaken at the end of, say one-half hour, the maximum pressure observed is the result of two variables, diastatic power and sucrose content. The sucrose, however,

has been completely fermented out before one-half hour, and increased pressure from that point on may be regarded as due solely to diastasis. Therefore, the differential between the values at the end of one-half hour (or possibly a still shorter interval of time) and at the end of 1 hour may be considered as a reliable index to the diastatic value of the flour. It is, of course, permissible to extend the time beyond one hour, although most flours failed to register any very large pressure increase after the 1 hour period. Flour 2, which was highest of all in diastatic power, showed an increase of 30 mm. pressure from 1 hour to  $1\frac{1}{2}$  hours, while flour 6 a low diastatic flour, under similar conditions showed an increase of only 4 mm. pressure.

There are obviously other purposes for which the manometric procedure herein outlined may be found useful. By substituting sugar solutions for flour suspensions, and decidedly reducing the quantity of yeast, the procedure may be found suitable for yeast testing. When the gassing power method was applied to standard sugar solutions of varying sugar content it was found that the maximum pressure developed is a linear function of the sugar concentration. This suggests that after calibrating the apparatus for known quantities of sugar the method may be used for the rapid elimination of sugar in solutions or extracts in which there is no diastatic activity. These possibilities remain to be investigated more extensively.

# Summary and Conclusions

1. A series of 33 representative baker's flours from various mills in the United States and Canada has been studied with reference to reducing sugar content, sucrose content, diastatic activity, and gassing power.

2. The quantity of reducing sugars present as such in normal, sound bread flours is extremely small (probably 0.1% to 0.2%) and nearly constant. Sucrose content is nearly 10 times as great, and its quantity was found to vary from 1.0% to 1.74%.

3. The blank determination in estimating diastatic activity by the Rumsey (1922) type of procedure is an unnecessary step for all ordinary purposes; either it may be dispensed with, or a constant correction for it may be applied.

4. A simple and apparently reliable manometric procedure for the estimation of gassing power of flour is proposed.

5. The terms diastatic activity and gassing power, as applied to flour, are not strictly synonymous. Lack of parallelism between these two properties is due to variations among flours with respect to original sucrose content.

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### HEATING OF FEED GRAINS

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#### Introduction

It has been shown by the work of many investigators that the spontaneous heating of cereal grains in storage is primarily the result of a respiration process. Under certain conditions, fermentations, mold growths, and intermolecular changes take place, but the main reaction which results in heating is a true respiration in which the grains absorb atmospheric oxygen and evolve carbon dioxide and water. The reaction is exothermal, the formation of a given weight of carbon dioxide being accompanied by the liberation of a definite quantity of heat.

Since grains respire continuously under all ordinary conditions it follows that they are always heating. Since they are poor conductors of heat, a rise in temperature may result if the *heating* is too fast. The heat energy released accumulates in the mass in proportion to its bulk. The rate of release of heat energy is ordinarily so slow that in a small bulk of grain there is no noticeable rise in temperature; the heat being lost to the air and surrounding objects as fast as formed. Under certain conditions, however, the rate of release may be so fast and the bulk so great that heat is not dissipated fast enough and a marked rise in temperature results.

The rate at which grains respire can be conveniently studied on a laboratory scale by the method of Bailey and Gurjar (Journal of Biological Chemistry, Volume 44, pages 5 to 17). By this method a small quantity of grain in a closed vessel is held in a thermostatically controlled oven for a given time, after which the respired carbon dioxide is determined. By proceeding in this way it can be shown that the rate of respiration is dependent upon several factors, the most important of which is the percentage of moisture in the grain. The rate is affected to a tremendous extent by a slight increase in moisture. As a result of tests made in this laboratory, grain at 72° F. was found to respire three times as fast when its moisture content was 15% as when it was 12%. Again, at 100° F. a sample with 19% moisture respired thirty-four times as fast as one with 10% moisture.

While the factors affecting heating of several of the more important whole cereals have been fairly well established in the laboratory, more detailed knowledge of these factors under warehouse and elevator conditions where much larger quantities of grain are handled does not appear to be available. This is especially true when the grains are milled into mixed feeds.

# Experimental

Apparatus: To determine under more practical conditions the effect of moisture upon the actual heating of grain in storage, a weather room, fitted with humidity and temperature control, has been constructed. In this room experiments are undertaken with respect to the heating of grains, livestock feeds, and manufactured cereal products, stored under semi-commercial conditions. From a commercial point of view it is highly desirable to establish moisture limits under which such products can be safely stored.

The temperature of the *weather* room is thermostatically controlled and can be easily held constant to within  $\pm$  1° C. The humidity is controlled by a whirling disc humidifier which also acts as a fan to maintain adequate air circulation. The relative humidity of this room

can be maintained constant to within  $\pm 2\%$ . A self-recording instrument keeps an accurate record of temperature and humidity.

Procedure: The grains and mixed feeds are stored in this weather room in 100-pound burlap bags. The lower layer of bags is supported 2 inches from the floor upon timbers, in the same manner in which grain and feed dealers are recommended to store their bags. The bags are piled seven high with about 1 foot of air space between each row.

The moisture content of the grain is adjusted by adding water to obtain initial moisture contents of 12%, 13%, 14%, 15%, 16%, etc. At least two bags of each moisture content and of each kind of grain to be tested are used. The corn used is cracked corn, while the oats are crushed in the manner that these grains are usually prepared for use in mixed feeds.

Each bag of grain or feed is fitted with a mercury thermometer with the bulb close to the center. These thermometers are read regularly every twenty-four hours. A bag temperature higher than room temperature is, of course, due to a heating reaction going on in the feed and is indicative of spoiling.

To date the room has been operated at temperatures of 60°, 90°, and 100° F., with a constant relative humidity of 70%. This humidity represents roughly the most unfavorable humidity continuously encountered anywhere in the United States.

The data obtained in these studies are too vast to be presented here in detail. Its nature, however, is illustrated by the data given in Tables I to VI, and Figures 1 and 2.

#### Oats

Series I: These investigations were carried on at 90° F. and 70% relative humidity. The detailed data are shown in Table I. It will be noted that although Table I indicates tendencies to heat at a moisture content of 14%, unquestionable and pronounced heating was evident at the 14.3% moisture level.

Series II: The investigation was repeated using a temperature of  $100^{\circ}$  F. Active heating was in progress at a moisture content of 14% and in a noticeably shorter time as can be seen from Table II.

#### Corn

Series I: In the instance of cracked corn stored at 90° F. and 70% relative humidity, noticeable heating began at a moisture content of 14.3% and increased in intensity as the moisture level became greater. The data in this connection are given in Table III.

Series II: Cracked corn stored at 100° F. and 70% relative humidity heated faster than when stored at 90° F. and 70% relative humidity,

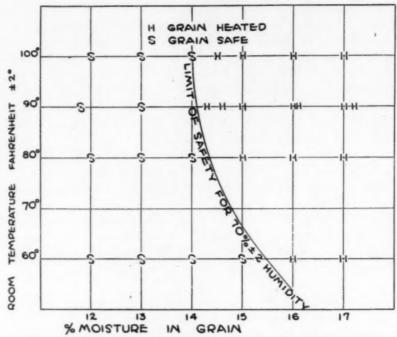


Fig. 1. Effect of room temperature and the moisture in the grain upon the heating of cracked corn.

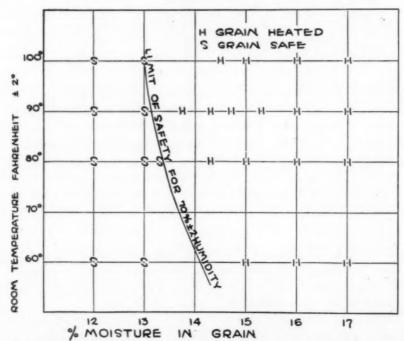


Fig. 2. Effect of room temperature and the moisture in the grain upon the heating of crushed oats.

TABLE I TEMPERATURE OF OATS STORED AT 90° F. AND 70% RELATIVE HUMIDITY

Time in storage				1	nitial	mois	ture co	ntent	of oats	in per	cent		
(days)	` 1	2	12	13	13	14	14.3	14.7	15.3	16	16	17	17
4	9	0	90	89	90	90	89	85	90	83	83	85	86
. 5	9	0	90	89	90	89	90	90	100	83	83	88	89
6	9	0	90	89	90	88	93	95	108	88	88	90	90
7	9	0	90	89	90	88	95	95	109	88	88	95	101
8	9	0	90	89	90	88	95	98	122	89	89	109	115
9	9	0	911	89	90	89	95	100	114	89	89	109	114
11	9	0	90	89	90	90	97	100	110	95	90	108	111
12	9	0	90	90	90	90	97	100	104	108	91	108	111
13	9	0	90	90	90	90	97	98	100	109	95	104	108
14	9	0	91	90	90	90	97	98	100	109	100	102	100
15	9	0	91	90	90	90	97	94	95	108	102	104	104
16	9	0	90	90	90	92	97	94	95	104	104	100	100
18	9	0	91	90	. 90	93	93	91	97	101	101	99	99
19	9	0	90	90	90	93	93	90	97	100	98	98	98
20	9	0	90	90	90	93	91	90	97	99	94	95	98
21	9	0	90	90	90	91	91	90	97	99	94	95	95
22	9	0	90	90	90	91	91	90	97	96	94	95	95
23	9	0	90	89	90	91	93	90	95	96	94	95	95
25	9	0	91	90	90	90	91	90	95	96	94	94	94
26	9	0	90	89	90	90	90	90	95	96	94	95	94
27	9	0	90	89	90	90	90	90	95	91	91	95	94
28	9	0	91	90	90	90	90	90	93	91	90	95	91
30	9	0	90	90	90	90	90	90	92	90	90	94	91
31	9	0	90	90	90	90	90	90	92	90	90	94	91

<sup>1</sup> Figures in italic indicate temperatures higher than the temperature of weather room.

<sup>2</sup> The initial low temperatures at the beginning of storage are due to the fact that the grains when transferred from a cold warehouse in winter weather to the experimental storage room required several days to reach the temperature of the room.

TABLE II TEMPERATURE OF OATS STORED AT 100° F. AND 70% RELATIVE HUMIDITY

Time of		I	nitial r	noistur	e conte	nt of o	ats in	per cen	it	
storage (days)	12.4	12.4	13	13.1	14	14	15	15	16	16
1	70 1	71	74	76	75	78	71	75	70	71
4	85	86	88	88	90	88	80	80	90	91
7	90	90	93	93	92	93	91	91	100	100
9	100	100	100	100	105 2	100	96	90	100	100
10	100	100	100	100	118	118	105	105	105	108
13	100	100	100	100	105	110	110	110	105	108
15	100	100	100	100	108	111	110	110	105	108
18	100	100	100	100	106	107	110	110	105	108
20	100	100	100	100	103	103	108	109	110	108
21	100	100	100	100	100	100	105	105	108	109
23	100	100	100	100	102	102	106	106	107	109
25	100	100	100	100	100	100	105	105	102	105
27	100	100	100	100	100	100	106	105	105	105
29	100	100	100	100	100	100	104	106	103	104
30	100	100	100	100	100	100	102	102	102	102

<sup>1</sup> See footnote 1, Table I. <sup>2</sup> See footnote 2, Table I.

TABLE III

TEMPERATURE OF CRACKED CORN STORED AT 90° F. AND 70% RELATIVE HUMIDITY

Time in storage (days)		Initial moisture content of corn in per cent													
	12	12	13	13	14.3	14.6	15	15.5	16	16	17	17			
4	89 1	85	83	83	81	86	86	86	88	88	83	85			
5	90	85	83	85	81	86	86	89	88	88	85	85			
6	90	85	85	85	81	86	86	95	89	89	85	85			
7 8	90	88	85	85	81	86	90	100	90	90	85	85			
8	90	90	85	85	81	86	92	100	92	90	88	89			
9			89	89	89	89			96	92	89	89			
10	90	90	-				105	104							
11	90	90	90	90	90	89	107	107	102	93					
12	90	90	90	90	90	90	108	108	104	100	90	90			
13	90	90	90	90	90	90	107	107	108	108	97	95			
14	90	90	90	90	90	90	104	104	108	108	110	104			
15	90	90	90	90	90	92	104	104	108	108	115	110			
16			90	90	90	94			104	104	115	115			
17	90	90	-				101	100			105	108			
18	90	90	90	90	963	98	101	101	106	104					
19	90	90			96	98	101	100	106	104	106	105			
20	90	90	89	90	100	100	101	100	104	102	106	106			
21	90	90	89	90	100	100	101	97	102	102	108	106			
22	90	90	89	90	94	100	100	97	100	101	106	106			
23	20		89	90	94	98			99	100	101	101			
24			-				99	95							
25	90	90	89	90	94	95	97	94	98	95					
26	20		0,		95	95	96	94	96	94	100	100			
27	90	90	88	88	95	95	- 0		96	94	100	100			
28	-0	-0	00		93	95	94	91	95	91		.00			
29	90	90	88	88	93	90			92	90	100	99			
30	90	90	89	89	91	90	90	90	91	90	95	94			

<sup>1</sup> See footnote 1, Table I. <sup>2</sup> See footnote 2, Table I.

TABLE IV

Temperature of Cracked Corn Stored at  $100^{\circ}$  F. and 70% Relative Humidity

Time of storage (days)	Initial moisture content of corn in per cent											
	12	12	13	13	14	14.5	14.7	14.8	16	16		
1	80 1	80	80	80	80	80	80	80	80	80		
4	85	85	87	88	88	87	88	86	86	85		
7	86	88	90	92	93	91	90	95	90	91		
9	100	100	100	100	95	95	95	95	95	95		
10	100	100	100	100	100	100	100	100	108	108		
13	100	100	100	100	100	100	100	103	108	111		
15	100	100	100	100	100	100	100	103	108	108		
16	100	100	100	100	100	100	102	105	108	108		
18	100	100	100	100	100	100	108	105	108	109		
20	100	100	100	100	100	100	110	105	108	108		
21	100	100	100	100	100	103 2	109	110	108	108		
23	100	100	100	100	100	104	107	112	106	107		
25	100	100	100	100	100	107	108	109	105	106		
27	100	100	100	100	100	107	108	108	105	105		
29	100	100	100	100	100	107	108	108	105	105		
30	100	100	100	100	100	107	108	108	105	105		

<sup>1</sup> See footnote 1, Table I. <sup>2</sup> See footnote 2, Table I.

as can be seen from a study of Table IV, indicating that when the initial temperature of storage is high the storage risk is increased.

#### Mixed Feeds

Series I: Mixed feeds constituted the material for the next investigation. The mixed feed in question happened to be a mixture of cracked corn, crushed oats, alfalfa meal, linseed meal, and molasses. In this series the temperature of storage was held at 90° F. and 70% relative humidity. Table V records data from these tests showing that heating began at a moisture content of 14.3%, but was not quite as active as was the case with corn and oats of the same percentage of moisture. However, at the high moisture levels heating was more intense than for corn and oats.

TABLE V
TEMPERATURE OF MIXED FEED STORED AT 90° F. AND 70% RELATIVE HUMIDITY 1

Time in storage (days)		Initial moisture content of mixed feed in per cent												
	12	13	13.4	14	14.3	15	15.5	16	16	17	17.5			
4	89 2	88	89	86	86	88	88	88	88	85	84			
5	89	88	89	88	89	88	88	89	88	86	84			
6	89	88	89	89	89	88	89	88	88	89	90			
6 7	89	88	89	89	89	88	89	88	.88	94	104			
8	89	88	89	89	89	88	90	88	88	108	120			
9	90	88	89	89	89	88	91	89	89	116	120			
10	90			89	89									
11		88	89			90	102	94	100	116	120			
12	90	88	89	89	89	93	106	104	111	115	118			
13	89	88	89	89	89	96	111	112	115	115	118			
14	89	88	89	89	89	100	108	115	115	116	118			
15	89	88	89	90	90	100	108	112	113	115	115			
16	89		-	90	90									
17		89	89		-	100	108	112	115	113	115			
18	89			90	90					111	115			
19	89	88	88	90	90	102	108	108	108	111	113			
20	89	88	88	90	90	107	109	108	108	111	113			
21	89	88	88	90	90	108	108	106	108	111	118			
22	89	88	88	90	92	109	109	106	108	111	115			
23	89	88	88	90	92	108	108	104	108	111	113			
24		88	88		-	108	108	104	104					
25	90	00	00	90	92	200	200	201		115	115			
26	90	88	89	90	92	108	108	104	108	113	113			
27	90	88	90	90	92	107	104		200	111	113			
28	90	88	90	90	92	107	106	104	108	111	113			
29	90	88	90	90	94	107	106	104	102	111	113			
30	90	88	90	913	96	107	106	104	102	119	111			

A mixture of cracked corn, crushed oats, alfalfa meal, linseed meal and molasses.

<sup>2</sup> See footnote 1, Table I. <sup>3</sup> See footnote 2, Table I.

Series II: The temperature of storage was  $100^{\circ}$  F. and the relative humidity 70%. These data are shown in Table VI.

TABLE VI
TEMPERATURE OF MIXED FEED STORED AT 100° F. AND 70% RELATIVE HUMIDITY

Time of storage (days)	Initial moisture content of mixed feed in per cent											
	12	12	13	13	14.5	14	14.7	14.7	16	16		
1	80 1	80	80	80	60	60	60	60	60	61		
4	85	85	88	88	75	75	74	75	70	72		
9	100	100	100	100	97	100	95	97	98	100		
10	100	100	100	100	100	100	100	100	100	100		
13	100	100	100	100	100	100	100	100	100	105		
15	100	100	100	100	100	100	100	100	110	110		
16	100	100	100	100	103 2	100	100	102	110	110		
18	100	100	100	100	103	100	106	106	110	110		
20	100	100	100	100	103	100	110	110	109	109		
21	100	100	100	100	103	100	109	108	110	110		
23	100	100	100	100	104	100	109	109	111	109		
25	100	100	100	100	106	100	106	108	106	108		
27	100	100	100	100	106	100	110	108	108	108		
29	100	100	100	100	106	100	110	110	109	109		
30	100	100	100	100	106	100	109	109	107	107		

<sup>1</sup> See footnote 1, Table I. <sup>2</sup> See footnote 2, Table I.

The same general picture is to be found when mixed feeds are stored at high temperatures, namely, that storage hazards are increased and that a moisture content in excess of 14.5% presents distinct storage risks.

#### Discussion

Figures 1 and 2 record the data obtained at 60° F. as well as 90° F. and 100° F. The points marked "S" indicate samples tested in the experiments which were not heating. The points marked "H" indicate samples that were heating. From the data shown in Tables I to VI, as well as from the supplementary data given in Figures 1 and 2, it is evident that crushed oats to be safe in storage must have a moisture content of at least 1% less than cracked corn for the same temperature. The curves also show that cracked corn or crushed oats held at a temperature of 100° F. must have 1% to 1½% less moisture, to be safe in storage, than the same grains at 60° F.

From the data presented, cracked corn did not begin to heat until its moisture content was slightly above 14%. At 15% heating was quite active. Cracked corn that contained 14% or less moisture has never been observed to be heating. The practical conclusion is that 14% should be the maximum allowable moisture content for corn to be used in mixed feeds.

Crushed oats show a slightly greater tendency to heat than cracked corn. A few samples of oats have been observed to be heating at a moisture content of 14%. For crushed oats  $13\frac{1}{2}\%$  moisture is considered the maximum allowable.

Molasses feeds, made mainly from cracked corn and crushed oats, with small quantities of other ingredients and running from 10% to 20% molasses, are popularly supposed to heat more easily than straight grains. However, the tests show that such feeds do not heat more readily than straight grains of the same moisture content. The poor keeping qualities of molasses feeds appear to be due entirely to the fact that they usually run too high in moisture. When dried to 14% moisture they may be carried in storage without fear of heating.

Observations made with corn grits, made by cracking corn and sieving out most of the germ, heat at a slower rate than cracked corn with the germ left in. In explanation, the enzymes affecting respiration are more abundant in the germ than in other parts of the grain.

Each grain and each grain product will have its own individual curve. Mixtures of grains, particularly with molasses, will develop a curve different from the sum of curves of the several ingredients. As a general rule, the finer the product is ground, the more easily will it go out of condition.

#### Conclusions

Cracked corn, crushed oats, and mixed feeds will not heat in storage when their moisture content does not exceed  $13\frac{1}{2}\%$ . They will heat if the moisture content is slightly above 14%.

### PRESIDENT'S ADDRESS

R. K. DURHAM

Rodney Milling Company, Kansas City, Missouri

(Read at the Convention, May, 1932)

We are assembled for our eighteenth annual meeting. That which has been accomplished in the past twelve months may be briefly reviewed and found to be of small magnitude, but the sum of the accomplishments of the past eighteen years is something about which we may modestly boast. The Association is carrying on by its own momentum and will probably continue to do so.

At this meeting considerable attention will be focused upon the report of the Committee on Standardization of Laboratory Baking. Cereal Chemistry of May, 1932, contains the final report of the laboratory activities of this Fellowship project. On Tuesday of this week, the entire morning session will be devoted to a discussion of the Research Fellow's activities. Some of you are probably disappointed because the final report does not outline minute specifications for performance and interpretation of the baking test. Sectionalism, commercialism, and personal feeling will be manifested in the discussion. It is to be hoped from the discourse will come guidance for future baking committees. Whether or not the work, up to this point, receives generous approval, one may safely predict that the data and observations of the Fellow will be of inestimable value to baking technologists. Of further interest in this connection is the fact that the Fellowship Fund established in 1929 is now exhausted.

The committee on Testing Soft Wheat Flour has been less favored. Although its work is perhaps broader in scope, it has been forced to depend upon whatever time its individual members could devote to the various projects undertaken. It was previously thought that a bread baking test could be evolved for soft wheat as well as bread wheat testing. Incorrectness of this assumption or delay in its confirmation has made it necessary for the soft wheat flour chemists to turn to other methods of testing. This Association should be on guard against the devotion of preponderous attention to any one type of flour.

An enormous amount of thought and collaborative study has been devoted to methods for the determination of ash, moisture, and protein in cereals and cereal products. It has been repeatedly demonstrated

that the methods adopted by this Association for these determinations can, with reasonable care and intelligence, be made to yield results which check within satisfactory limits. Previous Methods Committees have suggested that error to be expected between laboratories be defined.

It is emphatically urged that at this meeting this Association definitely place itself on record as to number of decimal places to be used in reporting ash, moisture, and protein, and the tolerance allowable between laboratories be defined.

At this annual meeting the Thomas Burr Osborne Medal will be awarded for the second time. It is not a prize to be greedily sought. The true scientist's reward is in the joy of discovery and service to mankind. This award to one of our own members is a commendable gesture of appreciation for what he has done for cereal chemistry and for the American Association of Cereal Chemists.

Inasmuch as a relatively small percentage of our entire membership is present at our annual meetings, and since so much time, that might otherwise be devoted to profitable discussion, is taken up by the present method of electing officers, it has been suggested that elections be by ballots sent through the mail to all members. It is recommended that the next Executive Committee consider the advisability of so amending the Constitution.

Upon his resignation from this Association, one prominent chemist voiced the opinion that the American Association of Cereal Chemists is fast tending toward a trade organization rather than a scientific one. Whether the accusation is justified or not, it should serve as a warning. A large majority of our members are employed by commercial interests. We must not allow the spirit of commercialism to overshadow our scientific thoughts and activities.

# REPORT OF THE COMMITTEE ON THE STANDARDIZATION OF LABORATORY BAKING

D. A. COLEMAN, Chairman

Bureau of Agricultural Economics, U. S. Department of Agriculture, Washington, D. C.

(Read at the Convention, May, 1932)

As was the policy of the Committee last year, the action of this year's Committee was confined to the development and approval of general plans and policies. This was a necessity as the widespread distribution of the personnel of the Committee made it impossible to hold meetings. However, much in the matter of constructive thought was acquired by correspondence, and such information was forwarded to the Baking Fellow and his associates for general use in furthering the objectives of the investigation. Under such a set-up it is again self-evident that direct responsibility for carrying out the details of the work was by necessity lodged in the hands of the head of the laboratory in which the studies were being conducted. In this respect, it is the belief of the Committee that Doctor Blish has shown excellent judgment in planning and carrying out the investigations which you have all had the opportunity to study by reason of its recent appearance in the May issue of Cereal Chemistry, and which we will shortly discuss.

Doctor Blish is to be further commended for assembling this fine report by reason of the fact that Mr. Merritt, the Research Baking Fellow, found it necessary to resign before all of the research data had been accumulated, and before any of the report had been placed in permanent form. With the aid of Mr. R. M. Sandstedt, whom the Baking Committee voted to place in Mr. Merritt's place for 30 days,

the report as printed was finally consummated.

On account of the wide difference in interests among the members of the A. A. C. C., as well as of certain interests who contributed to the Baking Fellowship fund, it was considered advisable to cover a wide range of topics rather than exhaustively study any one factor. Such a procedure, while not finally settling the problem in question, develops trends which are indicative of the value of the research at hand. Those who anticipated that the outcome of the Research Fellow's activities would permit a final establishment of specifications, both for performance and interpretation of the baking test, will doubtless be disappointed. It is the opinion of the Fellow and his associates that many years will elapse before this will be accomplished.

It is believed by the Baking Fellow and his associates, that the principal items of accomplishment are: (1) An improved knowledge and appreciation of the relative importance of some of the more conspicuous factors that affect both the baking test and its interpretation; (2) the securing of information as to the probable error of individual tests, and as to factors affecting variability; (3) modifications of procedure for special purposes and requirements; (4) the gaining of knowledge regarding types of equipment designed both for the convenience of the operator and for greater elimination of the personal element in manipulation; and (5) suggestions of appropriate subjects for future investigations.

It is hoped that all have taken the time to read the final report of the Research Fellow and that you are prepared to discuss it freely. We will proceed to a discussion of the report and recommendations of the committee.

#### Recommendations of the Committee

The committee recommends:

1. That the Hobart-Swanson mixer, with a mixing time of 1 minute, be considered as the official instrument and method for mechanical mixing.

2. That heavy tin pans be substituted for the 4X spotless metal

3. That stated supplementary procedure be eliminated. Any stated departure from the basic procedure to be considered as supplementary procedures.

4. That all other details of the basic procedure as originally set up are to remain the same.

It is further recommended that additional study and specific attention be given to:

(a) The shape of the baking pan.

(b) The place of the S-roll and other simple mechanical devices in relation to punching and molding as well as the possibility of entirely eliminating punching.

(c) That the so called "short method" with the elimination of sugar and 1½ hr. fermentation period be studied further.

(d) That additional effort be made to arrive at more definite oven specifications.

# REPORT OF THE COMMITTEE ON TESTING SOFT WHEAT FLOURS

MARY M. BROOKE, Chairman Purity Bakeries Corporation, Chicago, Illinois

(Read at the Convention, May, 1932)

After the adjournment of the 17th Convention at Louisville, last year, the Committee on Testing Soft Wheat Flours held a meeting and outlined this year's work in the form of subcommittees. Seven of these subcommittees were formed as the scope of the work of this general committee was too broad for any one person or one committee to cover the entire field. The subcommittee plan seems to have functioned satisfactorily.

No functioning chairman of any committee of this association can claim more whole-hearted support than the chairman of this committee. Every member of this committee has worked hard and effectively. A mid-year meeting was held in Detroit in November, at which time work accomplished by the various subcommittees up to that time was discussed and plans for the future made. The committee on Testing Soft Wheat Flours has been much more fortunate in formulating baking test methods for cake, pies, biscuits, and cookies than have the breadbaking committee. We have had no preconceived ideas to break down and have had to start from zero, pioneering in this work.

There will be one tentative baking test method and several proposed methods presented for discussion and action this morning.

The methods presented are not designed to make an acceptable baker's product. They are designed to evaluate flour for the purpose or use for which they are intended. They are formulated so as to eliminate as many variables as possible.

The Committee, after the several years' work, are unanimous in the decision that due to the physio-chemical and bio-chemical reactions involved in each type of product that no one test may be evolved that may evaluate flours for two or more purposes or uses. Consequently, the several methods proposed.

Each subcommittee chairman will present the results of his activities, after which the recommendations of the Committee as a whole will be given.

#### Recommendations of the Committee

1. You have heard and voted upon the various recommendations of the members of the committee. You can all realize the extremely broad scope of this work and the allied problems arising.

2. When this committee was first formed, it was designated as the Soft Wheat Testing Committee, or the Cake and Biscuit Flour Testing Committee. Its work was to cover all phases of the use of Soft Wheat Flour. This has not been changed since its inception.

3. As chairman of this committee, I have recommended for two previous years that the work of this committee be divided or subdivided *or* that the scope of the work be narrowed.

4. This year, I would like to say that it is physically and mentally impossible for any one person to efficiently handle the scope of this work. Consequently, I recommend to the incoming president that the scope of the work be narrowed, if necessary giving the committee a new name, or that the work be divided among several.

# REPORT OF THE SUBCOMMITTEE ON CAKE-BAKING METHOD FOR TESTING SOFT WHEAT FLOURS

## L. H. BAILEY, Chairman

Bureau of Chemistry and Soils, U. S. Department of Agriculture, Washington, D. C.

(Read at the Convention, May, 1932)

The committee has continued work along the line reported to the last convention. It was not possible for some of the members to attend a midyear meeting, hence most of the work had to be done by correspondence.

The aim of the committee has been to develop a method that will produce a white cake capable of showing differences resulting from variations in the character of the flours. To this end a number of modifications of the A. A. C. C. formula proposed in 1928, and also several other formulas specifying different proportions of constituents, were tried by the committee.

Many tests of these formulas have been made with a wide variety of soft wheat flours, and certain conclusions have been drawn.

It is the opinion of the committee that no one formula will make a satisfactory cake from every sample of flour. It is also realized that a flour may make a good cake with one formula handled under certain conditions and yet make a complete failure with another formula under the same or different conditions.

The committee realizes that making a white cake may not be the best way to test a flour which is to be used for pies, crackers, biscuits, or pretzels. At least it would require extensive experimentation and careful observation to be able to interpret the results obtained in making cake into terms indicating the suitability of the flour for making other baked goods. Nevertheless, it seems desirable for the Association to have some common means of measuring the relative values of soft wheat flours, and, therefore, the committee submits the following formula and method of procedure:

#### White Cake Formula

Shortening	65 gms.
Sugar	250 gms.
Flour .	260 gms.
Milk (skim)	230 gms.
(30 gms. skimmilk powder + 200 cc. water)	
Egg white	82 gms.
(12 gms. albumin + 70 cc. water)	0
Salt	2 gms.
Soda	3 gms.
Cream of tartar	6 gms.

Procedure: (1) Soak the albumin in water for at least one hour or preferably overnight in a refrigerator; (2) add the milk powder to the water and stir until the solution is complete; (3) dissolve the salt and soda in the milk; (4) sift the flour twice before using; (5) use electric mixer, equipped with 3-quart bowl, and having 3 speeds, approximate speed of beater as follows—low 120, medium 210, and high 380 r.p.m.; (6) place all ingredients, except cream of tartar, in mixer bowl; (7) mix at low speed until flour is no longer dusty, approximately 1 minute. Change to medium speed and mix for 10 minutes; (8) add cream of tartar and mix for 1 to 2 minutes; (9) use cake pans having approximately the following dimensions  $2\frac{1}{2}'' \times 3\frac{1}{2}'' \times 7\frac{1}{4}''$ . Use paper liners in the pans; (10) place 325 gms. of batter in each of 2 pans, without scraping bowl; (11) have all ingredients at about 70° F. before starting the mixer; (12) vary the quantity of milk to suit absorption of flour; vary time and temperature of baking to suit conditions of oven; (13) bake at 350° to 375° F. for 50 to 60 minutes; (14) cool at least 1 hour, preferably overnight before scoring.

#### Recommendations

The subcommittee recommends:

- 1. That the proposed method be adopted tentatively by the Association.
- That the members of the Association give this method a thorough trial and report their findings to the Committee on Cake Baking next year.
- That the method with modifications again be brought before the Association for adoption or rejection at the next convention.

## REPORT OF THE SUBCOMMITTEE ON CAKE SCORE-CARD

## R. A. BARACKMAN, Chairman Victor Chemical Works, Chicago, Illinois

(Read at the Convention, May, 1932)

The laboratory testing of flour for cake and biscuit making purposes has, in the past, been a matter of personal judgment, and must necessarily remain so. The committee has had no way to express to each other just what a particular flour will do in the baking, and to overcome this difficulty, the score card subcommittee has attempted to devise a means of such expression.

This year a questionnaire was sent out to 26 members of A. A. C. C. in which the 1931 tentative score card was submitted. Replies were received from 50% of these representing equally bakery, mill control, and research laboratories. These collaborators, no doubt, represent members most interested in the development of a score card, although only 4 have used the tentative 1931 system in their laboratories. Questions and replies will not be given in detail, but a brief summary of the ideas presented will be given.

The concensus of opinion was that the score card should be used for testing cake as cake when made from a particular flour. Some expression should be used to designate fat, sugar, and water tolerance, and these items should be expressed in terms of percentage of flour. The tolerance figures should be independent of the scoring system values, and preferably should be as addenda along with moisture, ash, and protein analyses.

The addition of figures to represent flavor and odor differences in the finished cake, particularly for commercial purposes, met with disfavor, and since the committee is only interested in the physical properties of flour which will produce a high quality baked product, the subcommittee recommends that flavor and odor be dropped from further consideration. Thus, two cards, one for chemists for judging flour, and another for judging commercial cakes should be used.

One collaborator neatly expressed the requirements of a score card as expressing, (1) the degree of perfection, and (2) remarks as to the faults. Final judgment should be based on cake score, i.e., perfection, on water tolerance, fat tolerance, sugar tolerance, and all other factors which enter into the quality of a flour and its ability to produce a high grade of cake.

Only 2 collaborators objected to the values assigned to individual properties of cakes as given in the 1931 score card. It was the judgment of the committee last year that these values are rational and indicate the relative weight on a basis of 100 total, which should be placed on each property as influenced by flour. All but one collaborator expressed the opinion that remarks should be recorded to indicate the fault and cause of low scoring, thus allowing a complete picture to be recorded for reference.

#### Recommendations

The subcommittee recommends:

1. That the values assigned in the 1931 score card (Cereal Chem. 8: 253) remain unchanged.

2. That fat tolerance, sugar tolerance, and water tolerance be designated in reports in terms of minimum and maximum per cent (flour basis) which will not impair cake quality.

3. That reports also include a statement of moisture, protein, and ash (calculated to 15% moisture basis), and hydrogen-ion concentration in terms of pH.

4. That viscosity values be added to reports dependent on the findings of the viscosity committee.

5. That final judgment of a cake flour be based on moisture, protein, ash, pH, tolerance figures, viscosity values and score with comments regarding faults.

## REPORT OF THE SUBCOMMITTEE ON PIE-FLOUR TESTS

C. B. Kress, Chairman

Sperry Flour Company, San Francisco, California

(Read at the Convention, May, 1932)

Large quantities of flour are used by pie factories for making pie crust. It is a highly specialized use of flour and the requirements are quite different from breadmaking. The qualities involved in a pie crust are color, tenderness, flakiness, form, and dryness.

Color should be a good brown. Some flours give a very pale appearing crust, which is not desirable. This quality is governed by the kind of wheat used and the bleach on the flour. Unbleached flour gives a browner color than bleached flour.

Flours vary much in the *tenderness* of the pie crust they will produce. Some will be very tough. Evidently this is governed by the amount and quality of the gluten. It is also possible to increase the shortening to overcome toughness.

Some flours give a *flaky* crust, while others give a mealy crust. A flaky crust is more desirable. This factor can be governed by the method of working, but it should be possible to produce a good flaky crust from a flour, particularly for the top crust.

Some flours hold the *shape* of the pie tin well, while others blister and swell. They should show a small amount of blistering, which indicates some strength in the gluten even after weakened by the shortening in the mixture.

With respect to dryness, it is best to examine pie shells the next day to observe this quality. This depends on the amount and quality of gluten and the absorption of the flour and shortening mixture. A good pie flour will make a pie shell that will keep dry, tender, and flaky. Poor quality pie flours make shells that become gummy and soft.

In addition to the above descriptive considerations, certain mechanical tests are also helpful in evaluating pie crust flours. A test which measures that portion of the crust that is readily mealy and granular has lately been developed in the Sperry Laboratories, and this test, as well as the descriptive tests listed above, have been used in the Committee work on the testing of pie crust flours. Aside from being a quantitative test, the test also shows up qualitatively, in a very dramatic fashion, differences in the quality of flours for pie crust manufacture. The greatest difference is found in the bottom portion of the crust.

TABLE I
CHEMICAL ANALYSES OF FLOURS USED IN THIS REPORT

Flours	Protein	Ash	Bleach	Absorption in bread	Volume 1-1b. Ioaf	Amount passing through no. 10 sieve	Absorption in pie crust
	P.ct.	P.ct.		P.ct.	.00	P.ct.	P.ct.
Family patent	10.5	0.38	Medium	09	2250	80	54
strong bakers	12.0	.40	Medium	62	2350	70	09
Clear from family	13.2	.72	Medium	62	2150	43	64
ancy cake flour	7.2	.36	Strong	100	1700	83	55
astry flour	7.2	.54	Unbleached	55	1800	91	50

TABLE II GRADING OF PIE CRUSTS BAKED FROM DIFFERENT FLOURS—AFTER 48 HOURS

Flour	Dryness	Color	Tenderness	Flakiness	Form
Family patent Strong bakers Clear from family Fancy cake flour Pastry flour	Slightly damp Damp Very damp Very dry Very dry	Brown Brown Very brown White Brown	Fair Tough Very tough Good Very tender	Good Compact Damp and heavy Very, very good	Good, blisters on bottom Good, some blisters Fairly good Regular Excellent, dry blisters

The experimental work for this paper consisted of making pie crust from various types of flour and then noting the physical qualities of the pie crust from each. The pie crust formula used was:

Flour	200 gms.
Shortening	100 gms.
Salt	2 gms.
Cold water	50-64%

Procedure: One-half of the shortening, all flour, and salt were mixed in a mixer until dry. This takes about 5 minutes. The remainder of the shortening is worked in by hand and finally the water is also mixed in by hand. Next, the pie dough is rolled out into a sheet  $\frac{1}{8}$  inch thick—cut out and pressed into a pie tin and baked with another pie tin laying on top to keep the pie dough down next to the tin. The shells are then baked in a moderate oven for about 15 minutes.

Mechanical test: After standing 24 hours the bottom crust of the pie shell was broken up by crushing, rubbing, and sifting through a No. 10 wire sieve.

The results of our efforts to evaluate different flours for pie crust purposes are shown in Tables I and II. Table I records the character of the flours used in the work, whereas Table II records the character of the pie crust obtained, using both the descriptive, as well as the mechanical test to score the material.

## Summary and Conclusions

From these tests it is to be concluded, (1) that pie crusts require special laboratory tests; (2) that from a careful examination of the bottom crust of pie shells, the quality of a flour for this purpose is best observed.

A mechanical sifting test is proposed which indicates pie crust flour quality which is both qualitative and quantitative in character.

## REPORT OF THE SUBCOMMITTEE ON TESTS FOR BISCUIT AND SELF-RISING FLOURS

L. D. WHITING, Chairman Ballard & Ballard, Louisville, Kentucky

(Read at the Convention, May, 1932)

In the examination of biscuit and self-rising flours, it has come to be generally conceded that the real test of flour is the character of the baked product that the flour is capable of producing. Bailey (1923) pointed this out and proposed a baking formula specifying 227 gms. of self-rising flour and 6.6% of shortening. He baked seven biscuits at 240° C. for 12 to 15 minutes.

A survey of a number of the published baking formulas for biscuits shows many variations in the formulas and in the procedures, and formulas now in use in laboratories would undoubtedly reveal similar variations. Jacobs (1922) used 250 gms. of flour and only 4% of shortening. He baked seven biscuits for 12 minutes at 500° F. Chittick and Dunlap (1925) proposed that biscuits be baked in a set of rings using 226.8 gms. of flour and 12.4% shortening. Baking conditions were given as 400° to 450° F. for 15 to 20 minutes. Only five biscuits were measured. Alexander (1925) used 227 gms. of flour and approximately 10% shortening; skimmed milk instead of water. The amounts of flour and shortening were reduced to round numbers by Logue and Ranker (1926) when they specified 200 gms. of flour and 15% of shortening. They cut five biscuits according to the Chittick and Dunlap method, and baked them at 475° to 500° F. for 15 minutes.

In all of these procedures the excess dough was discarded. As this amounted to more than half of the dough made, a reduction in the weight of flour sample to 125 gms. was suggested tentatively for this work. The amount of shortening used in many of the formulas mentioned was too meager, and consequently, should be increased sufficiently to work satisfactorily. Sixteen per cent of shortening is the amount suggested. Milk instead of water is proposed as the liquid. Generally speaking, the time of baking should be shorter than that specified in the procedures mentioned.

## Experimental

BAKING TESTS

A tentative formula and procedure for baking biscuits embodying these suggestions was used as follows: Self-rising flour Shortening Milk 125 gms. 20 gms. 80 cc.-85 cc.

Procedure: The shortening is mixed into the flour with the finger tips. Add 80 cc. of milk and mix thoroughly. Place the dough on a lightly floured board and shape by hand. Roll the dough ½ inch thick and cut it into seven biscuits 1¾ inches in diameter. Bake 8 to 10 minutes at 500° F. Cool and weigh at end of 30 minutes. Measure volume by seed displacement, considering each set of seven biscuits as a unit.

The amount of milk suggested is 80 cc. This may be varied slightly and in case of some flours may be 85 cc. The amount used should be recorded.

In the case of plain flour, 1.87% of monocalcium phosphate, 1.50% of sodium bicarbonate, and 2% of salt, based on the weight of flour, should be thoroughly mixed with the flour before adding the shortening.

In order to try out this tentative formula, samples of two flours, labeled A and B, both in plain and in self-rising form, were tested by the writer and Mr. H. G. Walter, whose assistance is gratefully acknowledged, and were baked and measured according to the above procedure. Flour A was a soft wheat flour with a protein content of 8.25% and an ash of 0.35%. Flour B was a hard wheat flour with a protein content of 9.75% and an ash of 0.45%. In addition to a study of the above formula, investigations were also undertaken in order to observe the effect of the thickness of doughs on the specific volume obtained, and separate tests were run on dough  $\frac{3}{8}$  inch,  $\frac{1}{2}$  inch, and  $\frac{5}{8}$  inch in thickness.

The results of the collaborative tests will be found in Tables I and II. It is to be noted from a study of the data in these tables that in most instances, the specific volume of the biscuits made from dough 5/8 inch in thickness was smaller than that obtained from 1/2 inch or 3/8 inch doughs.

Dough  $\frac{5}{8}$  of an inch thick is much too thick, and gives unsatisfactory biscuits in other respects. The  $\frac{1}{2}$  inch dough gives biscuits of the best appearance.

The weight of flour used in this test yields just seven biscuits 1¾ inches in diameter and ¼ inch thick without using the scrap. In a standard test, the weight may possibly be increased slightly to allow a sufficient though not excessive margin of dough.

Due to the elasticity of the dough, the dough will be more than  $\frac{1}{2}$  inch thick if the rolling pin is allowed to rest on a hoop  $\frac{1}{2}$  inch in depth. It was found that a hoop approximately  $\frac{7}{16}$  inch in depth was necessary to give a dough  $\frac{1}{2}$  inch thick.

TABLE I
RESULTS OF COLLABORATIVE BAKING TESTS AS SUBMITTED BY L. D. WHITING

				Flour A					Flour	ur B		
		Plain		S	Self-rising	186		Plain		S	Self-rising	18
Thickness of dough (inches)	3/8	1/2	00/	Using 80 cc.	milk 1/2	8/8	3/8	1/2	8/8	3/8	1/2	8/8
Amount of milk (cc.)	80	80	80	80	80	80	80	80	80	80	801	808
Weight of 7 biscuits (gms.)	66	123	156	101	138	167	86	132	155	104	133	169
Volume of 7 biscuits (cc.)	245	315	355	260	340	410	240	290	340	250	315	370
Specific volume, biscuit basis	2.47	2.56	2.28	2.57	2.46	2.46	2.45	2.20	2.19	2.40	2.37	2.19
				Using 85 cc.	milk							
Thickness of dough (inches)	3/8	1/2	5/8	3/8	1/2	5/8	3/8	1/2	5/8	3/8	1/2	5/8
Amount of milk (cc.)	85	82	82	85	82	82	85	82	80	00	82	00
Weight of 7 biscuits (gms.)	107	127	153	103	129	158	106	127	157	114	150	184
Volume of 7 biscuits (cc.)	265	335	390	265	330	410	255	310	355	255	355	410
Specific volume, biscuit basis	2.48	2.64	2.55	2.57	2.56	2.59	2.41	2.44	2.26	2.24	2.37	2.23

TABLE II
RESULTS OF COLLABORATIVE BAKING TEST AS SUBMITTED BY H. G. WALTER

			Flo	lour A					Flo	lour B		
		Plain		S	elf-rising	ng		Plain		S	elf-risi	ng
Thickness of dough (inches)	3/8	1/2	5/8	3/8	1/2	5/8	3/8	1/2	5/8	3/8	1/2	5/8
Amount of milk (cc.)	82	85	85	85	82	85	06	06	06	06	06	90
Weight of dough of 7 biscuits (gms.)	119	145	173	135	147	170	128	150	177	138	150	169
Weight of 7 biscuits (gms.)	105	130	157	121	132	154	114	135	161	124	136	154
Oven loss (gms.)	14	15	16	14	15	16	14	15	16	14	14	15
Volume of 7 biscuits (cc.)	227	269	318	273	292	336	263	291	345	264	294	334
Specific volume, dough basis	1.91	1.85	1.84	2.02	1.99	1.98	2.05	1.94	1.95	1.91	1.96	1.98
Specific volume, biscuit basis	2.16	2.07	2.03	2.26	2.21	2.18	2.30	2.15	2.14	2.13	2.16	2.17

Walter found it necessary to use an increased amount of liquid due to the fact that the samples seemed to be dried out slightly. This indicates the need for a specified moisture basis in weighing the flour sample.

There is a difference in the specific volumes reported by the two collaborators. This may be due either to oven conditions, or to the need for standardizing volume apparatus, or to the mixing conditions.

A set of blocks, measured in both laboratories, gave results which eliminated the volume apparatus as the cause of the difference. Subsequent correspondence has shown that the mixing conditions were not the same in the two laboratories. The need for the study of mechanical methods of mixing is, therefore, evident.

#### GASOMETRIC TESTS

One of the tests frequently used in testing self-rising flours is the determination of carbon dioxide. The gasometric method for CO<sub>2</sub> in baking powders has been developed to a high degree of accuracy. The use of a similar method for CO<sub>2</sub> in self-rising flour is likewise of great value.

The writer in the capacity of Associate Referee for the A. O. A. C. has recently engaged in collaborative work with respect to the determination of CO<sub>2</sub> in self-rising flours. The following material is taken from the report of the Associate Referee as submitted to the A. O. A. C. in November, 1931.

The method which was used in the collaborative work referred to is as follows.

#### REAGENTS

(a) Dilute sulfuric acid (1 + 5) by volume.

(b) Displacement solution: Dissolve 100 gms. of sodium chloride crystals in 350 cc. of water. Add approximately 1 gm. of sodium bicarbonate and 2 cc. of methyl orange indicator and then sufficient of the dilute sulfuric acid (1 + 5) to make just acid (a decided pink color). Stir until all carbon dioxide is removed. This solution is used in the gas-measuring tube and leveling bulb and seldom needs to be replaced.

#### Determination

The factor weight of self-rising flour, that is, ten times the weight of one liter of carbon dioxide at the temperature and atmospheric pressure obtaining at the time the tests are made, is weighed out and placed in the dry decomposition flask A 2 together with about two

See table for density of CO<sub>2</sub>, A. O. A. C. Book of Methods, 3d edition (1930), pages 556-562;
 S. W. Parr, J. Am. Chem. Soc. 31: 237 (1909).
 Refers to lettering on Figure 15, page 119, A. O. A. C. Book of methods, 3d edition (1930).

dozen glass beads the size of a pea, which break up any lumps formed by the flour when the acid solution is added. The flask A is then connected with the apparatus by means of the two-hole rubber stopper. The T tube stopcock (C) is opened and by means of the leveling bulb (E) the salt solution in the gas burette (D) is brought to the zero mark. The T tube stopcock is closed and 45 cc. of dilute sulphuric acid (1+5) are introduced into the decomposition flask from the acid burette (F). The level of the salt solution in the leveling bulb should at all times during the decomposition be kept at a slightly lower level than that in the gas measuring burette, to prevent the liberated carbon dioxide from escaping through the acid burette into the air, while the acid is being introduced. For the final reading, the salt solution in the gas burette must be level with that in the leveling bulb.

After the acid has been added, the flask should be agitated vigorously for about five minutes. The volume of gas liberated is then read and the apparatus allowed to stand for about ten minutes, at which time another reading of the gas evolved should be made. If these readings coincide, the volume of gas may be calculated. If some gas has been evolved during the time the apparatus was at rest, the decomposition flask should be further agitated and similar readings taken thereafter. When no change in the volume of gas is noticed on standing, all of the gas may be assumed to have been liberated.

In calculating the percentage of gas evolved, the number of cubic centimeters of acid introduced must be substracted from the final reading of the salt solution level in the gas measuring burette. The resulting difference may be taken as the volume of gas evolved. When the factor weight of self-rising flour is used, each cc. of gas represents 1/10% by weight of carbon dioxide.

Three samples of self-rising flour carefully prepared from airdried flour, salt, sodium bicarbonate and monocalcium phosphate, were sent to eight collaborators who reported on each sample in triplicate. A comparison of the theoretical CO<sub>2</sub> content of the flours with the average obtained by the collaborators is given in Table III.

The average result for each flour is lower than the theoretical. This difference may be due to two factors, either the loss of some of

TABLE III

RESULTS OF COLLABORATIVE TESTS TO DETERMINE THE CO<sub>2</sub> CONTENT OF SELF-RISING FLOURS

Sample	Theoretical CO <sub>2</sub>	Reported CO2	Recovery
	P.ct.	P.ct.	P.ct.
A	.746	.713	95.5
В	.867	.819	94.4
C	.982	.921	93.8

the gas by chemical action, or the retention of some of the gas by the flour-acid mixture in the flask.

The last named factor is apparently of more importance than the former. Further work is being done to find whether this retention is constant for all flours and whether it varies with the per cent of carbon dioxide present.

A comparison of the results obtained by the various collaborators shows only a fair amount of agreement. On samples B and C the maximum and minimum results are too widely divergent, differing from the average by 4%. Further work is being done to eliminate much of this error from the method.

#### Recommendations

The subcommittee recommends the following for future study:

- 1. The use of 200 gms. of flour on a specified moisture basis.
- 2. The use of mechanical equipment for mixing biscuit doughs.
- 3. Development of a method and apparatus for determining the volume of biscuits.
  - 4. Development of a score card for evaluating biscuits.
  - 5. Collaborative baking tests making use of the score card.

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## REPORT OF THE SUBCOMMITTEE ON TESTING OF SOFT WHEAT FLOURS FOR USE IN COOKIES

PEARL BROWN, Chairman Perfection Biscuit Company, Fort Wayne, Indiana

(Read at the Convention, May, 1932)

The soft wheat flours supplied to biscuit and cracker manufacturers are used in a variety of products, each of the units of which has quite distinctive characteristics. Roughly these products are divided into two groups, namely, sponge goods and sweet goods, or crackers and cookies.

To make clear the scope of the word cookie, a classification might be of value. The one given was compiled by A. A. Schaal. classification the cookies are grouped according to the machine upon which they are formed, namely:

1. Cutting machine varieties

a. Panned cakes Honey cake type Molasses cake type Sugar cake type Lemon crackers Fruit bars Embossed English type

b. Peeled varieties (baked on oven shelf)

Graham crackers

2. Bar press types Fig bars Taffy bars, etc. 3. Wire cut (soft cakes) Honey cake type Molasses cake type Ginger snap Vanilla wafer type Macaroons

4. Deposit—(soft cakes) Vanilla wafer type Molasses cake type Jumble and fancy design type

5. Dutch cookie machine cakes

6. Sugar wafers

Each of the machines indicated handles doughs of a certain character; and the products made therefrom are different from those made upon the other machines.

The characteristics of these various cookie doughs are again quite different from those doughs made and rolled out by hand, as is the practice in shops where machines are not used, where the cookies made are of the home-made type.

## Experimental

In seeking to evaluate a flour for use in cookies, the complete picture should be considered so that the final results obtained may be interpreted into actual shop practice.

The easiest way to observe whether or not differences exist between flours is to bake them. Various test-bake formulae have been used in studying the effect of the various ingredients used in cookies.

Because the formula for plain cookies (sugar) is quite easily modified to produce other types of product, it was arbitrarily taken as a basis for study. Several formulae—both experimental and shop, were tried. It was found that either type of formula indicated differences in flour when all conditions were maintained the same and where the flour was the only variable ingredient.

The following simple formula was, therefore, used to start these investigations:

Flour	226 gms.
Sugar	113 gms.
Shortening	75 gms.
Milk (dry skim)	33/4 gms.
Eggs (dry whole)	2 gms.
Salt	$1\frac{1}{4}$ gms.
Cream of tartar	1 gms.
Soda	$\frac{1}{2}$ gms.
Water	q.s. (65 cc.)

Mixing Procedure: The shortening and all dry sifted ingredients were placed in the machine and mixed one minute on slow speed. The water was added during the next twenty seconds, and the mixing continued forty seconds. The dough was cut down, mixed another minute on slow speed, cut down, and mixed twenty seconds on second speed. The temperature out of the mixer was 75° to 76° F. The dough was rolled out by hand on a board equipped with rails so that all cookies were of uniform thickness. The cookies were cut out by hand and baked at 425° F.

Three phases of study were to have been considered, (1) the effect of flour grade, (2) the effect of bleach, and (3) the value of viscosity measurements.

Effect of Flour Grade: In studying the effect of grade, a series of flours from two different mills were used. Admittedly, this is not sufficient samples from which to draw general conclusions, but as the results obtained check the results for the same study with other types of flour, the results no doubt would point to the same conclusions.

In this series of tests, 4 flours, a 20%, a 50%, an 80%, and a 90% flour, milled from Indiana wheat were used. The analysis of the flours is given below:

Flour p.ct.	Moisture p.ct.	Ash p.ct.	Protein p.ct.	pH of bleached sample when used
20	13.10	.30	5.98	5.3
50	13.10	.32	8.74	5.3
80	13.00	.36	8.91	5.4
90	12.90	.39	9.23	5.45

The color, grain, texture, and spread of the cookies were better baked from the short patent flours than from the 80% and 90% flours. Differences, however, were not so great as were expected.

In the second series of tests, flour was obtained from a different mill. This flour consisted of a 20%, a 50%, and an 80% flour, milled from a mixture of Indiana and Michigan wheat.

The analysis of these flours follows:

Flour	Moisture	Ash 1	Protein 1	pH	
p.ct.	p.ct.	p.ct.	p.ct.	Unbleached	Bleached
20	11.00	.31	7.90	5.9	5.5
50	10.82	.35	8.24	5.9	5.5
80	10.88	.38	8.50	5.9	5.6

<sup>1 15.0%</sup> moisture basis.

Cookies baked from this series of flours were practically the same as for the previous series. The spread, grain, texture and general eating qualities of the cookies were best baked from the shorter patent flours, but the differences again were not great.

As would be expected, the crumb color of the cookies made from flours of shorter extraction was better than that of the cookies from the longer patents. Crumb color, of course, is not of so much importance in most cookies as it is in soft cakes.

To summarize, in both series of flours tested, the flours of shorter extraction produced cookies with the best characteristics; the characteristics considered being external color, spread, grain, texture, and tenderness. It may be, however, that these differences would be more distinct in soft doughs. That was not tried because of lack of laboratory equipment for the uniform depositing of soft doughs.

Effect of Bleaching: The other question considered was the effect of bleaching the flour. For this study, the two series of flour, just described, were used, as well as separate samples from other mills.

In the first series of tests, the flour was obtained unbleached, and bleached with Novadel-Agene. In every case in this series of tests, the cookies baked from the unbleached flour had greater spread than had the cookies from the bleached flours. The pH of the 20% flour was 5.3; of the 50% flour, 5.3; of the 80% flour, 5.4; and of the 90% flour, 5.45. This was taken colorimetrically after the flour had stood in the laboratory for several weeks.

In the second series of tests the flour was bleached with Novadel only. In these tests, the bleached flours gave cookies with noticeably better spread. The difference, however, was not quite as pronounced as in Novadel-Agene bleached flours. To confirm this, a new sample of flour from this same mill and the same wheat mix was obtained. This time the sample was divided as follows: One part was unbleached, one part was bleached with Novadel only, and one with Beta Chlora only. Again the Novadel bleached flour gave the best spread. The Beta Chlora bleached flour gave greater spread than the unbleached flour, but less than the Novadel bleached flour.

One sample of flour milled from a mixture of red and white Michigan wheat (50% of each) was used. It was obtained unbleached and bleached with Novadel and chlorine. The pH of the bleached sample was 5.45. The analysis was:

Moisture	14.00%
Protein	8.76%
Ash	.38%

The bleached flour produced a cookie with less spread than that produced by the unbleached flour. Both cookies, however, had very good characteristics. The flour was very soft.

## Summary

(1) In one series of flours milled from Indiana wheat, flour bleached with Novadel-Agene produced cookies with less spread than unbleached flour. (2) With a second series of flours milled by a different mill, from a mixture of Indiana and Michigan wheat, flour bleached with Novadel alone produced cookies with a better spread than did the unbleached flour. (3) Other samples of flours milled from the same wheat blend, bleached with Beta Chlora produced cookies with better spread than those made from unbleached flour, but with not as much spread as those cookies produced from Novadel bleached flour. (4) A fourth flour, milled from a mixture of red and white Michigan wheat, bleached with Novadel and chlorine to a pH of 5.45, produced cookies with less spread than the same flour unbleached.

#### Recommendations

- 1. That the effect of the various bleaching agents commonly used be studied,
- (a) upon the same soft wheat flour, and
- (b) upon flours of the same extraction from different soft wheat mixtures.
- 2. That a record of the pH of the flours and the doughs made therefrom before baking be kept.
- 3. That a study be made of the value of the viscosity measurement in evaluating cookie flours.

## REPORT OF THE SUBCOMMITTEE ON HYDROGEN-ION CONCENTRATION WITH SPECIAL REFERENCE TO THE EFFECT OF FLOUR BLEACH

## E. E. SMITH, Chairman F. W. Stock & Sons, Hillsdale, Michigan

(Read at the Convention, May, 1932)

During the past year L. E. Sleeper, of our laboratory, who did most of the work, and to whom indebtedness is hereby gratefully acknowledged, and I have completed a preliminary and entirely sketchy survey of the general effect of chlorine bleaching upon certain soft wheat flours, as revealed by the test cake of our Association.

The single-stage method of mixing was employed throughout; the formula used being as follows:

Hydrogenated shortening	60 gms.
Sugar, finely granulated	200 gms.
Flour	250 gms.
Dry skimmed milk	15 gms.
Albumen	15 gms.
Salt	3 gms.
Cream of tartar	4 gms.
Soda	2 gms.
Water	190 cc.

This formula, it will be noted, differs from the modification recommended by L. H. Bailey in that it carries more albumen and less leavening. As reported last year, it was found, in the case of our oven at least, that the albumen content of the formula had to be increased in order to secure a cake that did not break in two under the crust; and with this increased amount of albumen it was found that 4 gms. of cream of tartar produced the best cake.

For the purposes of this study various streams and combinations of streams of flour were taken; two friction-top tin cans being filled with identical sub-samples of each flour. The contents of one can of each sample was treated with chlorine in an experimental bleacher, secured through the courtesy of the Wallace and Tiernan Company, to a pH of approximately 5.4.

Each sample was analyzed and baked, the results secured being shown in Tables I and II which are self-explanatory, except for a description of the various flours. The first letter identifies the sample, the second tells whether or not it was bleached—C meaning chlorine, U

TABLE I

RESULTS OF CAKE BAKING TESTS USING BLEACHED AND UNBLEACHED FLOUR FROM VARIOUS FLOUR STREAMS

Flour	H2O	Ash	Pro- tein	Vis- cosity 1	pН	Parts chlorine	Cake volume	Cake
	P.ct.	P.ct.	P.ct.				cc.	
AC					5.3	10	840	951/2
AU	14.53	0.309	6.19	15°			855	95
BC					5.2	11	910	98
BU	13.43	0.327	7.71	39°			865	97
CC					5.25	10	900	971/
CU	13.35	0.330	7.91	38°			870	961/2
XC1				46°	5.0	20	810	97
XC2				49°	5.4	12	885	98
XCA				48°	5.4	-	860	99
XU	13.55	0.330	8.65	52°			850	97
DC					5.25	12	895	98
DU	14.43	0.338	7.71	34°			875	97
EC					5.2	20	890	98
EU	14.53	0.346	6.07	14°			845	96
FC					5.35	10	870	97
FU	13.55	0.352	7.31	30°			870	96
GC					5.3	11	825	96
GU	12.98	0.354	9.30	66°			830	951/
HC					5.3	11	895	971/
HU	14.53	0.361	9.34	33°			870	961/
IC					5.35	12	850	961/2
IU	12.78	0.373	9.14	54°			860	951/2
JC					5.3	12	870	98
JU	14.62	0.374	8.55	35°			870	97
KC					5.4	12	830	96
KU	12.55	0.385	9.98	78°			830	951/
LC					5.4	13	810	95
LU	12.30	0.407	9.82	75°			810	941/2
MC					5.35	12	910	98
MU	14.10	0.410	8.59	$40^{\circ}$			920	97
NC					5.5	15	910	97
NU	13.80	0.427	9.82	38°			930	97
OC					5.4	17	800	95
OU	12.18	0.467	10.74	110°			825	95
PC					5.4	21	890	97
PU	13.00	0.476	8.07	25°			900	961/
OC					5.4	24	850	971
QU	13.77	0.522	8.43	21°			870	961/
RC					5.45	35	790	96
RU	12.08	0.571	9.82	52°			830	95
SC					5.35	40	860	97½ 96½
SU	13.15	0.590	9.30	24°			910	961/
TC					5.35	45	640	90
TU	12.73	0.606	11.02	71°		4.0	780	91
UC					5.4	48	700	91
UU	12.20	0.710	10.66	30°	- 45	***	780	911/
VC				200	5.45	50	710	93
VU	13.13	0.873	10.98	30°			760	92

Degrees Mac Michael.

meaning unbleached, and A meaning Agene. The flours are arranged in ascending order of ash content.

Inasmuch as there is considerable variation in the manner with which viscosity of flour and water suspensions is determined, the

#### TABLE II

Symmetry and Internal Characteristics of Loaves Baked from Flours Listed in Table I

Flour	
AC	Symmetrical, grain rather coarse.
AU	Flatter and coarser, slacker.
BC	Symmetrical, fine; very few small holes.
BU	Flatter and coarser; somewhat slacker.
CC	Symmetrical, fine; few small holes.
CU	Flatter, coarser; but holes smaller.
XC1	Symmetrical, grain very fine, quite a few medium holes.
XC2	Symmetrical, very fine, few holes.
XCA	Symmetrical, very fine, very few holes.
XU	Symmetrical, rather coarse grain, many small holes.
DC	Symmetrical, very fine, few small holes.
DU	Somewhat flatter and coarser, but excellent.
EC	Symmetrical, very fine, very few small holes.
EU	Flatter, coarser, more and larger holes.
FC	Symmetrical, fine, few small holes.
FU	Flatter, coarser, more and larger holes.
GC	Not well rounded, grain fair, several medium holes.
GU	Flatter, coarser larger holes.
HC	Symmetrical, fine, few small holes.
HU	Flatter, coarser, larger holes.
IC	Symmetrical, fine, several large holes.
IU	Flatter, coarser, larger holes.
IC	Symmetrical, fine, very few small holes.
IU	Flatter, coarser, holes somewhat larger.
KC	Symmetrical, 4-8 large holes per slice, 1 bad tunnel.
KU	Flatter and coarser, holes about same.
LC	Symmetrical, rather coarse, 6 holes per slice, crust pale.
LU	Symmetrical, coarser, more and larger holes, crust pale.
MC	Symmetrical, fine, few small holes.
MU	Somewhat flatter, coarser, but fewer holes.
NC	Symmetrical, good grain, 3 large holes per slice.
NU	Flatter, coarser, fewer large holes.
OC	Batter viscous, symmetrical, coarse, 2-4 large holes, pale.
OU	Batter viscous, symmetrical, coarser, 2-4 large holes, pale.
PC	Salvy, slack, symmetrical, fine, many small holes.
PU	Salvy, slack, symmetrical, coarser, holes larger.
OC	Symmetrical, fine, few small holes.
ÕU	Flatter and coarser, more and larger holes.
ŘC	Symmetrical, fine, many small holes.
RU	Flatter and coarser, fewer but larger holes.
SC	Symmetrical, fine, very few small holes.
SU	Flatter and coarser, holes about same.
TC	Tough, sogg_', ragged; bad tunnels and holes.
TU	Not quite so soggy and ragged; tunnels and holes about same.
UC	Tough, soggy, ragged; tunnels and holes; pale.
UU	Somewhat better, ragged; tunnels and holes; pale.
VC	Stiff and viscous; ragged, soggy, tunnels, holes; pale.
VU	Stiff and viscous; flatter, coarser, soggy, more but smaller holes; pale.

following is a description of the apparatus and procedure used in obtaining the values given in Table I.

Apparatus. MacMichael viscosimeter, large bowl, flat bob, No. 30 wire, speed adjusted to exactly 12 r.p.m. by stopwatch. Be sure that pointer reads exactly zero when bob is hanging free in saddle.

Porcelain mortar, inside diameter  $12\frac{1}{2}$  cm. best size, with well-fitted pestle.

Temperature. Room temperature for routine work, but for more precise results temperature should be controlled to some definite standard—we use 25° C.

Method. Weigh out 20 gms. of flour, to within 2 mg. Correction for moisture will be necessary if variation from standard is much over 1%. Measure out in graduated cylinder 100.0 ml. distilled water of high grade. (Poor quality distilled water will give low results.) Pour about 90 ml. of this water over the 20 gms. of flour in the mortar. Grind with pestle lightly but rapidly until smooth suspension is obtained. After some practice, not more than 45 seconds should be required for this grinding. Pour suspension into bowl of viscosimeter. Rinse mortar and pestle with remaining 10 ml. water, with a little light grinding. Add rinsings to main portion of suspension in viscosimeter bowl. Pipette 5.0 ml. Normal lactic acid (factor anywhere between 0.99N and 1.01N is close enough) into suspension in viscosimeter bowl. Stir thoroughly with bob of viscosimeter, hang bob in saddle, start machine (damping first rapid swing by touching disk very lightly with tip of finger), and read displacement on scale as soon as equilibrium is attained.

#### Discussion of Results

Score. It is seen that chlorine treatment of these flours increased the score of cakes baked therefrom in almost every instance. The only flours of which the unbleached samples produced higher-scoring cakes than the bleached—T and U—were streams of very poor quality, never used in cake work. In fact, the cakes produced from these flours, as well as those from flour V, also of very poor quality, were so poor that the scores applied to them are practically meaningless; both bleached and unbleached.

Volume. In general, and especially in the flours of higher quality, chlorine bleaching increased the loaf volume significantly. In the middle grades the volumes tended to be approximately equal, while in the lower grades chlorine treatment definitely lessened the cake volume. It may be that this lessened volume is due to the heavy dosages required to overcome the buffering action of the flours in question—needing, as they did, four or five times as much chlorine as did the flours of lower ash content.

Symmetry. In almost every case chlorine bleaching improved the symmetry of the test cake.

*Grain.* In every case, down even to the poorest flours, chlorine treatment improved the grain of the test cake.

#### Comments

The work described here is only a preliminary study. It has, however, developed a number of points which will serve as guides in future work. Future work should consist of a more intensive study of a limited number of representative flours; each treated to varying degrees of pH, with and without aeration, and with and without combinations of different oxidizing and bleaching agents. Such advanced work has not been started, for two reasons; first, because of lack of time, and second, because it seems to the writer that the formula and method used herein do not reveal differences between certain flours which commercial experience has shown to vary significantly under shop conditions. It is hoped that this problem may be attacked with greater vigor during the coming year, and with weapons of greater power than those now available to the subcommittee. hoped that my fellow subcommittees have perfected not only a formula that will work and that will reveal small differences, but also a scorecard that will really score.

## REPORT OF THE SUBCOMMITTEE ON VISCOSITY

## G. L. ALEXANDER, Chairman

This report was given extemporaneously and will appear as part of the stenographic record of the proceedings of the Convention which are to be published later by the Association.

#### REPORT OF THE COMMITTEE ON METHODS OF ANALYSIS

C. E. MANGELS, Chairman

North Dakota Agricultural Experiment Station, Fargo, North Dakota

(Read at the Convention, May, 1932)

A report of the principal activities of this Committee will be given in separate papers by C. F. Davis and A. E. Treloar. Davis has made a study of the magnesium acetate rapid ash procedure, while Treloar has made further studies on accuracy of routine chemical methods.

A year ago, the Secretary-Treasurer reported that the first edition of the Book of Methods was entirely sold, and it was suggested that this committee consider the matter of issuing a revised edition. To this end, some questionnaires have been sent to members of the Committee, and others, in order to determine what the membership desires in a revised edition. As one member of the Committee has observed, the field of the cereal chemist is constantly expanding, and a compilation of methods which would be satisfactory to the chemist today must cover a larger field than the previous volume. A number of new sections or chapters to the Book of Methods have been suggested, among which the following may be mentioned:

(1) Baking Procedures, which would include bread, cake, and biscuit methods; (2) Leavening Agents, which would include both yeast and chemical leavening agents; (3) Limits of Accuracy of Chemical Procedures, including various mathematical formulae used for computation; (4) Bake-Shop Ingredients other than Flour, Particu-

larly Shortenings.

Similarly, a number of additional reference tables have been suggested. These include (1) Munson-Walker table for converting Cu<sub>2</sub>O to sugar; (2) Latest N-protein factors based on work of D. B. Jones; (3) Vitamin table which would list briefly the different vitamins and their presence in different foods; (4) Table listing properties of commonly used indicators; (5) Table for determining relative humidity from wet and dry bulb readings; (6) Table showing composition of milk in different forms used by bakers; (7) Table giving composition of different dried egg preparations; (8) Table giving the CaO and P<sub>2</sub>O<sub>5</sub> content of feeding stuffs; (9) Table for converting voltages to pH values; (10) Table giving concentration of common laboratory reagents.

From comments received, it also appears that a very general opinion prevails that the section on *Bacteriological and Similar Methods* could be considerably improved.

Several have suggested the desirability of including the Bunzell test for rope, and one Committee member suggests directions for a germination test on wheat to determine the percentage of dead kernels.

With respect to additions and revisions in chemical methods, from the questionnaire it is evident that the cereal chemist of today is concerned with rapid procedures other than those for moisture, protein, and ash. There is, in particular, a demand for a rapid, accurate, volumetric procedure for sugars.

Other suggested methods for study include:

1. The inclusion of additional sugar methods. Munson-Walker, Schoorl's method, the Schafer-Hartman method, and the picric acid colorimetric method, have been suggested.

An acidity method, based on alcoholic extraction, similar to the Greek method.

3. Amplification of electrometric H-ion method and the inclusion of a colorimetric procedure.

4. Protein methods: (a) Allow use of metallic mercury and copper as catalysts. (b) State definitely proportion of Hg and Hg precipitating agents. (c) Revision of methods for determining different nitrogen fractions.

5. Method for determining original ash in phosphated and self-rising flour.

6. Qualitative test for phosphated and self-rising flours.

7. CO<sub>2</sub> test for chemical leavening agents.

8. Method for determining staleness in bread.

9. Kreiss test for rancidity.

10. Rask method for starch.

11. Tests for various bleaching agents and bread improvers.

With regard to the make-up of the Book of Methods, it is believed that this could be improved and particularly should be improved as regards cross-references. The majority opinion is that a permanent binding rather than the loose-leaf type is desirable.

The next edition of the Book of Methods should, therefore, cover a larger field than the present volume. The present time is probably not propitious for publishing a new edition even if a manuscript fully covering the needs of the cereal chemist were ready for the printer. The present edition is exhausted but the A. O. A. C. has recently issued a new edition of the well-known volume "Official and Tentative

Methods of Analysis." This new edition contains a much improved cereal section.

#### Recommendations

The committee recommends:

- 1. That a definite moisture basis be adopted for reporting chemical constituents of wheat, flour, and feeds.
- 2. That a similar moisture basis be adopted for reporting water absorption of flours.
- 3. That the Association recognize the limits of accuracy for protein determinations, for the determination of moisture by the oven method, and for determination of ash in flour.
  - 4. That the single filtration procedure for crude fiber be abandoned.
- That the alcoholic extraction or Greek acidity method for flour acidity be further studied.
- 6. That methods available for sugar analysis be studied, with a view of standardizing one or more procedures.
- 7. That methods of analysis for chemical leavening agents be studied.
- 8. That methods for the analysis of baking materials such as malt extracts, milk and egg products, shortening, etc., be studied.
- 9. The magnesium acetate alcohol rapid method for ash for mill control work.
- 10. That the printing of another edition of the A. A. C. C. Methods of Analysis be delayed until such time that a thorough and critical revision of its material can be completed.

# REPORT OF COLLABORATIVE STUDIES ON THE MAGNESIUM-ACETATE-ALCOHOL QUICK ASH PROCEDURE 1

C. F. DAVIS

Western Star Flour Mills, Salina, Kansas

(Read at the Convention, May, 1932)

In most methods of chemical analysis, time is an important factor. A reduction in the time allotted for the analytical procedure may mean that less accurate results will be obtained. Coleman and Christe (1925) found that the most reliable ash results on all samples of flour were obtained by burning the charge for 16 hours, at 585° C. There are many instances in the cereal industries where a great reduction of the ashing time, or a reduction of the three to five hour period for

This paper was read as a subcommittee report of the Committee on Methods of Analysis.

ashing, as used in many laboratories, would mean a greater usefulness for the laboratory. This is particularly true in flour mill control where the ash content of the flour is made an important factor.

Spaulding (1930) proposed the use of magnesium-acetate-alcohol in connection with the use of oxygen as a means of rapidly ashing flours. However, in the procedure <sup>2</sup> which has been given coollaborative study, the use of oxygen in the furnace has been substituted for by use of a platinum dish accompanied by a longer ashing period and a higher temperature of combustion. Eliminating the use of oxygen simplifies the procedure while the time remains within the time necessary to make a moisture test, thereby giving the analyst an ash test to a specified moisture basis in a short time. The original Spaulding procedure has been further modified by weighing the ash directly on the balance pan.

There are many difficulties to overcome in ashing flour. Walters (1930) in discussing new aids in ashing flour, outlines some of them. With these in mind, five flour samples of widely different characteristics were sent to a number of selected collaborators, who were equipped to give this quick procedure a fair trial. A questionnaire was also sent to each collaborator to obtain his reaction to the method. A summary of the numerical reports should give information as to how well the outlined difficulties could be met, particularly as to whether the inorganic radicals were fixed at this high ashing temperature. Johnson and Scott (1928) found that in ashing flours above 750° C. there was a definite loss in ash weight. Should this submitted method fail to prevent this loss, or should the loss be greater with some types of flours than others, one type of flour for a blank determination might not give a MgO factor suitable for various flours. This fact also should manifest itself from the collaborative reports.

## Experimental

## OUTLINE OF PROPOSED METHOD

Dissolve 15 gms. of magnesium-acetate C. P. in denatured alcohol, making the solution up to 1000 cc. (20° C.). Allow the solution to stand overnight and filter if the solution is not clear.

A 3'gm. sample of flour is weighed into a flat platinum crucible and an accurate 3 cc. portion of magnesium-acetate-alcohol solution is pipetted evenly over the flour surface, being sure that all the flour is wet.

The crucible and contents are then placed in a muffle at 850° C. and allowed to flame until all of the combustible material has burned.

<sup>&</sup>lt;sup>2</sup> This procedure was submitted to the Methods of Analysis Committee by W. L. Heald, of the Laboratory Service Division, Commander-Larabee Corporation, Kansas City, Missouri.

The muffle door is then closed. The crucible and contents are allowed to remain in the muffle until complete incineration or constant weight is assured. This time usually takes from 30 to 45 minutes. The crucibles are then taken from the muffle and placed in a dessicator. Only a short time is needed for the platinum to cool. When sufficiently cool, the crucible is tapped gently with a spatula to loosen its contents, which is dumped directly on balance pan and weighed.

With each set of quick ash tests a standard flour should be run, in which the ash content has been determined by long burning, in order to establish a correction for the blank. A blank of about 0.0085 to

0.0090 gm. seems to be the proper one.

It is best not to scrape the inside of the crucible, since gently tapping on the bottom will usually deposit all the ash on the balance pan. After each determination, the crucibles should be brushed thoroughly with a camel's hair brush.

#### Material Used

Flour A, a flour made from Bluestem and early Baart wheats for use in the manufacture of crackers. Submitted by Mr. Geo. H. Buford, Globe Grain & Milling Co., Ogden, Utah. Moisture 12.5%, protein 11.2%. Flour B, a spring wheat flour for bakery use. Submitted by Mr. Leslie R. Olsen, International Milling Co., Minneapolis, Minn. Moisture 12.4%, protein 15.0%. Flour C, a winter wheat flour for bakery use. Submitted by Mr. C. F. Davis, Western Star Mill Co., Salina, Kans. This flour was the same as the December check sample sent to the Pioneer and New York Sections of the A. A. C. C. Moisture 11.9%, protein 11.4%. Summary of Pioneer Section Report: 47 collaborators, mean ash content .4284%, standard deviation .0056%, C. V. 1.30%. Flour D, a short patent soft wheat flour submitted by Mr. C. M. Estling, Pillsbury Flour Mills, Springfield, Ill. Moisture 11.0%, protein 7.7%. Flour E, a durum wheat flour-a sample of durum semolina was submitted by a macaroni manufacturer and was ground to the fineness of very granular flour on an experimental mill. Moisture 12.3\%, protein 12.6\%. Each sample was thoroughly mixed for one hour in a mechanical mixer.

#### Results of Collaborative Tests

From the mean ash content (Table I) by the collaborators regular method (R) and the magnesium-acetate-alcohol method (M), one cannot say that one method gives a significantly higher or lower yield than the other. As the standard deviations and coefficients of variation are greater on each sample for method M, this indicates a greater variation of results in the group of collaborators by the quick procedure.

TABLE I

ASH CONTENT OF WHEAT FLOUR SAMPLES SENT OUT FOR COLLABORATIVE STUDY (R) Collaborators Regular Method. (M) Magnesium-Acetate-Alcohol Method

To the same of the	Collaboratore	Sam	Sample A	Sam	Sample B		Sample C	Sam	e D	Sam	Sample E
	Collaborators	Method R.	Method M	Method	Method M	Method R	Method M	Method R	Method	Method	Method M
		P.ct.	P.ct.	P.ct.	P.ct.	P.ct.		P.ct.	1		P.ct.
1	Mary M. Brooke	0.475	0.472	0.462	0.459	0.429	0.412	0.364	0.339	0.629	0.636
7	The W. E. Long Co.	.454	.447	.455	464	.423	.425	349	342	809	614
n	L. R. Van Auken	.460	.460	.465	.470	435	440	350	360	625	615
9	Mark	.470	.470	.470	.480	.440	.433	.360	377	643	019
		.467	.480	.463	.480	.433	.430	363	353	630	623
1	F. A. Collatz	.466	.470	.466	.468	.435	.438	.370	.366	.623	.631
00	Commander-Larabee Corp.	.450	.456	.451	.455	.425	.427	.344	.357	.610	.618
6	A. D. Wilhoit	.460	.480	.460	.465	.420	.430	.355	.350	.610	.615
10	Earl C. Paulsel	.457	.458	.455	.441	.430	.416	.352	.346	.617	.598
11	Marjorie Howe	.460	.450	.470	.470	.430	.430	.360	.350	.620	.620
12	L. G. Crandall	.450	.455	.453	.455	.417	.413	.333	.340	.610	.603
13	C. F. Davis	.458	.457	.465	.466	.432	.430	.350	.353	.614	.617
14	J. B. Merryfield	.453	.450	.460	.467	.427	.425	.357	.357	.610	.610
15	H. A. Baehr	.455	.452	.465	.463	.432	.418	.363	.338	.616	.613
16	G. A. Davis	.450	.451	.459	.455	.420	.421	.347	.348	.610	.613
17	C. M. Estling	.464	.462	.468	.462	.428	.428	.354	.354	.616	.615
200	H. H. Johnson	.455	.465	.477	.479	.427	.450	.345	.355	.615	.618
20	R. M. Sandstedt	.460	.457	.470	.477	.433	.423	.380 1	.393 1	.620	.623
21	M. D. Mize	.450	.446	.460	.447	.420	.433	.355	.350	.620	.621
22	Joe Stoklas	.451	.453	.465	.467	.430	.430	.340	.341	.603	.607
23	Paul McSpadden	.457	.460	.460	.470	.430	.427	.345	.350	.611	.613
24	C. E. Mangels	.470	.480	.480	.470	.440	.430	.350	.350	.620	.620
25	H. C. Fellows	.456	.458	.458	.468	.432	.433	.352	.348	.611	.614
26	L. H. Bailey	.470	.460	.480	.480	.430	.430	.360	.340	.620	.640
27	R. M. Knudson	.450	.450	.450	.450	.420	.420	.343	.343	.617	.617
Vumber	of samples	25	25	25	25	25	25	24		25	25
fean yie	Mean yield Standard deviation	.4587	.4599	.4634	.4651	.4287	.4276	.3525	.3502	.6171	.6169
ariation	Variation coefficient	1.59	2.17	1.72	2.19	1.30	2.20	2.41		1.23	1.45

Note: No report received from collaborators No. 3, 4 and 19. 1 Not included in calculations.

Vote: No report received from collaborators No. 3, 4 and 19, 1 Not included in calculations.

The comparison of the ratios of the differences between the means and standard deviations to their respective probable errors, which are given in Table II, gives figures to compare with the analysis made by Treloar (1928) on the collaborative ash study made by the 1927 Methods Committee where the analysts used their regular ashing

#### TABLE II

Comparison of the Ratios of the Differences Between the Values for R and M to Their Probable Errors

(R) Collaborators Regular Ashing Methods. (M) Magnesium-Acetate-Alcohol Ashing Method Values Expressed in %

		Mean Ash	Content		
Sample	Mean yield	ls R and M	Difference	$E_{(\overline{R}-\overline{M})}$	$\overline{R} - \overline{M}$
number	Method R	Method M	R - M	L(H-M)	$E_{(\overline{R}-\overline{M})}$
A	.4587 ±.0009	.4599 ±.0013	+.0012	.0016	+0.70
B	$.4634 \pm .0010$	$.4651 \pm .0013$	+.0017	.0017	+ .98
C	$.4287 \pm .0007$	$.4276 \pm .0012$	0010	.0014	70
D	$.3525 \pm .0011$	$.3502 \pm .0012$	0022	.0016	-1.32
E	$.6171 \pm .0010$	$.6169 \pm .0012$	0001	.0015	10

#### Standard deviations of ash content

Sample	Star	ndard de	viations	$\times$ 10 <sup>6</sup>	Difference	$E(\sqrt{M}-\sqrt{R})$	$\sqrt{M} - \sqrt{R}$
number	er Method R		Met	hod M	$\sqrt{\mathrm{M}} - \sqrt{\mathrm{R}} \times 10^6$	×106	$E(\sqrt{M}-\sqrt{R})$
	$\sqrt{R}$		$\sqrt{M}$				
A	7318	$\pm 698$	9997	$\pm 953$	2678	1188	2.25
В	8014	$\pm 764$	10230	±975	2216	1238	1.78
B	5590	$\pm 533$	9428	士899	3838	1030	3.72
D	8515	$\pm 828$	8960	$\pm 872$	445	1202	.37
E	7625	$\pm 727$	9004	$\pm 858$	1379	1124	1.22

method and the glycerol-alcohol ashing method. While the averages of these figures show a similar degree of variability between the two methods studied in each case, when special consideration is given to sample "C" in the present study, the average variation of the five samples involved is slightly less than found in the former study with the glycerol-alcohol method. It will be observed that the standard deviation of the regular method of ashing sample "C" (0.0055%) is low and can be considered close to a minimum value for collaborative ash analyses. This statement is based on the standard deviation figures obtained in the analysis of the 1927 report as worked out by Treloar and the records of the monthly check sample report of the Pioneer Section. In this last report 1607 ash tests were made on 46 different flour samples. For the four year period (1928 to 1931 inclusive) the average standard deviation was 0.0080%. Consistent results were also obtained with sample "C" when submitted to the Pioneer Section

as the December 1931 collaborative sample. At the time the standard deviation was 0.0056%. This is possibly a characteristic of this particular sample and if the regular method ash results had been less consistent or nearer the average, a comparison of the ratios between the difference in the values and their probable errors, by the two methods, would have been less significant.

When a direct comparison is made between the relative variability of the five samples studied, we find sample "E" to show the lowest variability (Coefficient of variation—method R, 1.23%; method M, 1.45%) and sample "D" the highest variability (Coefficient of variation—method R, 2.41%; method M, 2.55%) of results and sample "C" falls closely in line with the others. The figures show that sample "E" gives a definitely lower degree of variability than sample "D" by both ashing methods, this indicating a difference in ashing characteristics of the samples which affects the relative variability of results in collaborative ash reports to an extent as great as the differences in variability between the two methods studied. The slightly higher relative variability in the results reported on all samples by the magnesium-acetate-alcohol method is well in line with what we would expect with the increased possibility for error introduced by the quick method.

In Table III will be found a comparison of ash results by the regular and magnesium-acetate-alcohol methods as reported by members of the Northwest Section A. A. C. C. on one of the Sections regular collaborative flour samples.

TABLE III

NORTHWEST SECTION A. A. C. C. COLLABORATIVE REPORT

Flour Sample No. 164

C-11-1		Ash content
Collaborator	Regular method	Magnesium-acetate alcohol-method
	P.ct.	P.ct.
Marjorie Howe	.51	.51
A. D. Wilhoit	.51 .51	.505
F. A. Collatz	.51	.51
J. W. Whitacre	.50	.496
Earl C. Paulsel	.50	.50
Chas. W. Ingram	.49	.494
Average	.503	.503
Average (27 collaborators)	.500	

## Collaborative Opinion of the Method

Collaborators comments and suggestions taken from the answers to a questionnaire were as follows:

Seven were strongly in favor of using only an indirect weighing of the residue to prevent *blowing* of the ash. They reported the results of an indirect weighing as they found the direct weighing too inaccurate.

Six were experienced operators of the magnesium-acetate-alcohol ashing method. The experience of the others was limited to the necessary preparation for giving the proposed method a fair trial.

Five did not use flour in determining the MgO factor, but evaporated and ignited 3 cc. of the magnesium-acetate-alcohol solution. Three found considerable variation in the blank. Four did not find it necessary to run a blank with each ash determination as the MgO factor was relatively constant when carefully determined for a particular solution.

Three obtained the results they reported, by using porcelain crucibles. One found silica and porcelain dishes to give fairly dependable results when new, but found that they depreciated rapidly and could not recommend their use at the high temperature specified. One used a nickel-alloy wire as a counter-balance for the MgO factor. Two suggested the use of 13.5 gm. magnesium acetate per liter, to give a slightly lower blank. Three found a small camel's hair brush useful in transferring the ash from the dish to the balance pan. Three found the blank to vary with different types of flour. None found the hygroscopicity of the residue to affect the ash weight any more than this factor affects the weight of a normal flour ash.

A large majority of collaborators using their own slight modifications of procedure, were of the opinion that this proposed quick ash method was very dependable for mill control when carefully performed.

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#### MINUTES OF THE EIGHTEENTH ANNUAL CONVENTION OF THE AMERICAN ASSOCIATION OF CEREAL CHEMISTS

M. D. Mize, Secretary-Treasurer Statler Hotel, Detroit, Michigan

May 23-26, 1932

#### Monday, May 23

Convention called to order at 10:00 a.m. by President R. K. Durham. Invocation by the Reverend B. J. Bush of the Westminster Presbyterian Church.

A message of welcome was delivered by J. Lee Barrett, executive vice-president

of the Detroit Convention and Tourist Bureau.

Communications and greeting were received and read from the following: R. Wallace Mitchell, Leslie R. Olsen, C. W. Partridge, secretary Association of Operative Millers, Chas. Armuth, Newton C. Evans, E. S. Stateler, C. W. Brabender, and Dr. Ir. G. Van Der Lee.

The following committees were appointed by President Durham: Nominating committee: M. J. Blish, Chairman, C. G. Harrel, J. A. Dunn. Auditing committee: V. E. Fisher, Chairman, E. F. Tibbling, R. A. Barackman. Resolutions committee: Paul Logue, Chairman, Pearl Brown, A. G. Olsen President R. K. Durham delivered the address given on pages 402 to 403 of this issue of Cereal Chemistry.

A short report of the Committee on Osborne Medal Award was given by M. A. Gray announcing that the second award of the Medal would be made to C. H. Bailey

at the Monday evening meeting.

Meeting adjourned at 11:45 a.m.

Meeting called to order at 1:30 p.m. by President Durham. Meeting was placed in charge of Washington Platt, chairman for the afternoon. Paper—"The Testing of Flour by Means of the Recording Dough Mixer" by C. O. Swanson and E. B. Working, read by E. B. Working.

Paper—"Soft Winter Wheat Studies: A Whole Wheat Meal Viscosity Test for Detecting Certain Types of Poor Milling Wheats" by E. G. Bayfield.

Paper—"A Discussion of the Meaning of Some Terms Used in Cereal Chemistry" by Quick Landis and C. N. Frey, read by Quick Landis.
Paper—"An Automatic Method for Measuring the Expansion of Doughs" by

C. H. Bailey and Max C. Markley, read by C. H. Bailey.

Paper—"Regional and Seasonal Variation in Pigmentation of Durum Wheats" by C. E. Mangels.
Paper—"Statistics in the Service of Cereal Chemistry" by A. E. Treloar.

Meeting adjourned at 5:15 p.m.

A joint meeting of the Detroit Section of the American Chemical Society and the American Association of Cereal Chemists, preceded by a dinner at 6:30 p.m. was called to order at 7:30 p.m. by Harvey M. Merker, Chairman of the Detroit Section. After giving a short message of greeting to the members of the American Association of Cereal Chemists, Mr. Merker introduced President Durham. President Durham introduced C. B. Morison who delivered the Presentation Address of the Thomas

Burr Osborne Medal to Clyde H. Bailey, second recipient of this reward. President Durham then presented the Medal to Dr. Bailey.

President Durham then introduced C. H. Bailey as the speaker of the evening who delivered the following paper, "A Quarter Century of Cereal Chemistry." Meeting was adjourned by Mr. Merker.

#### Tuesday, May 24

Meeting called to order by President Durham at 9:30 a.m. Report of the Committee on Standardization of Laboratory Baking by D. A. Coleman, Chairman.

M. J. Blish then conducted a discussion of the "Report of Activities of A. A. C. C. Baking Research Fellowship" by P. P. Merritt, M. J. Blish and R. M. Sandstedt as printed in Cereal Chemistry, Vol. IX, No. 3, May 1932, page 175 to 238.

Pearl Brown moved that the report of the Committee on Standardization of Laboratory Baking and the published report of the Fellow and associates be accepted. Seconded, carried.

Meeting adjourned at 12.00 noon.

At 8:00 p.m. C. G. Harrel showed motion pictures entitled "The Eyes of Science," compliments of Bausch and Lomb. He also showed pictures taken by himself of baking reactions and procedures and also pictures taken at the 1931 Convention.

#### Wednesday, May 25

Meeting called to order by President Durham at 9:30 a.m.

Messages of greetings were read from H. H. Trapp, president of the Association of Operative Millers, Robt. L. Gertes of the American Society of Bakery Engineers. An invitation to our members to stop at Battle Creek on Friday, May 27th, to take part in the meeting of their local section of the American Chemical Society and to visit the Kellogg plant was extended by H. K. Wilder. Also, an invitation was read from M. S. Fine to visit the Postum and General Foods Corporation plants at Battle Creek.

Meeting placed in charge of Mary M. Brooke, Chairman for the morning.

P. E. Minton moved that a telegram of condolence be sent to D. L. Boyer on the occasion of the death of his mother. Seconded, carried.

Report of the Committee on Testing Soft Wheat Flours by Mary M. Brooke,

Chairman.

Sub-report by R. T. Bohn, "Standard Cake Baking Test." L. H. Bailey was chairman of this sub-committee and R. T. Bohn delivered the report in the absence of L. H. Bailey.

Sub-report by L. E. Lackson and Rufus Barackman, read by Barackman, "Score Card."

A. W. Meyer moved that the score card recommended be approved. Seconded, carried.

Sub-report by C. B. Kress, "Pie Flour." Because of a serious illness C. B. Kress could not be present and the report was read by J. A. Dunn.

J. A. Dunn moved that the recommendation of this report be accepted. Seconded, carried.

Sub-report by L. D. Whiting, "Biscuits and Self-Rising Flours."

L. D. Whiting moved that the recommendations made in his report be accepted. Seconded, carried

Sub-report by Pearl Brown, "Crackers and Hard Biscuits."

Pearl Brown moved that the recommendations in her report be accepted. Seconded, carried.

Sub-report by G. L. Alexander, "Viscosity."

G. L. Alexander moved that the recommendations in his report be accepted. Seconded, carried.

Sub-report by E. E. Smith, "The Hydrogen-ion in Test Cake."

E. E. Smith moved that the recommendations in his report be accepted. Seconded, carried.

Meeting adjourned at 12:15 p.m.

Meeting called to order by President Durham at 1:30 p.m.

C. E. Mangels moved the Minutes of the 1931 Convention by M. D. Mize, secretary-treasurer be approved as printed in Cereal Chemistry, Vol. 8, No. 4, page 335. Seconded, carried.

C. H. Bailey moved that the Annual Financial Report of the Secretary-Treasurer for the year 1931 be approved as printed in Cereal Chemistry, Vol. IX, No. 3, page 325. Seconded, carried.

#### Report of Editor-in-Chief

#### D. A. Coleman

Only one or two matters of importance have come to the attention of the Editor of Cereal Chemistry during the past year. There is an insistant demand on the part of many that there be published more papers of the so-called "practical nature." Assuming that the Editor was able to obtain such papers, which by the way is not

easy, raises the question of what kind of "practical paper" is to be recognized as

suitable for Cereal Chemistry.

I am quite sure that each and all of us do not want Cereal Chemistry to accept articles for publication which should rightfully find their place in trade journals; yet, on the other hand, there are certain types of laboratory findings that do lend themselves to presentation in our journal.

The other matter that has had my attention is the use of trade names in journal articles. I would like to hear a discussion of this matter, as with some of us the use of a trade name, even from a non-advertizing standpoint, is provocative of

much discussion and resentment.

E. N. Frank moved that the report of the Editor-in-Chief be accepted. Seconded, and carried.

#### Report of Managing Editor

#### C. C. Fifield

The financial statement of the Secretary-Treasurer published in Cereal Chemistry, Volume 1X, No. 3, shows the balance of the journal account at the close of the year December 31, 1931. This statement shows that a \$200.00 increase of funds were granted by the Executive Committee in September to help defray the expenses incidental to moving office equipment at St. Paul to Washington, D. C., and expressing 10,000 back issues of the journal to Lancaster, Penna. A portion of this fund was also used to bind in blue buckram enough volumes to supply the needs of those requesting back volumes to complete their files.

I regret that the outlook for advertising in the journal is not as favorable as it was some two years ago. However, I wish to record here my appreciation of the support of those advertisers who are patronizing Cereal Chemistry and would most

sincerely urge that they receive every consideration from our readers.

May I also ask for your personal solicitation of advertising for the journal because I believe that a word coming from you coupled with our own efforts will be most effective.

The Managing Editor still has for sale, in addition to back volumes, complete files of the journal beginning with Volume I, unbound, but the policy established some years ago of not selling certain issues singly must be adhered to, as the supply of some of the issues, especially those printed in 1924, is exceedingly small.

Compared with a year ago, there is a slight decrease in the number of subscribers and members which will equal in round numbers about 40. This is not alarming, however, but I must remind you that the editors are using every available means to utilize the funds to the best advantage so that the same high standard of quality and workmanship as well as the size of the journal are continued.

M. A. Gray moved that the report of the Managing Editor be accepted. Sec-

onded, carried.

#### Report of the Executive Committee

#### L. D. Whiting, Chairman

During the past year, the Executive Committee of the Association has carried on the routine business which has been assigned to it. The Chairman has approved the bills and countersigned the checks issued by the Secretary-Treasurer. Over 100 checks totaling more than \$7,000.00 have been countersigned. In this connection, the Secretary-Treasurer M. D. Mize, deserves much credit for the efficient manner in which he had conducted his office. He has in this way greatly simplified the financial duties mentioned.

Last year, the Executive Committee suggested the appointment of a Committee to examine the possibility of defining geographical boundaries and minimum membership of the local sections. The Committee under the Chairmanship of Bert Ingels has reported. The Executive Committee approves the recommendation in his report, namely, that we should not limit the membership or the areas of the local sections. The Executive Committee, however, wishes to bring to the attention of the local sections the fact that sections must comply with the provisions of the Constitution that all officers and at least three-fourths of the members of such organization shall be active members of the American Association of Cereal Chemists.

The Executive Committee last summer approved the transfer of \$200.00 from the Association account to the Cereal Chemistry account to cover partially the expense of moving the Cereal Chemistry properties to Washington.

The Committee approved the extension of time of the Baking Fellowship to

The Committee has approved the acceptance in 1932 of foreign members' dues and subscriptions in foreign money, but recommends that 1933 invoices specify

On account of the banking situation in some sections, approval has been given to the plan of deposition immediately in a Washington bank the checks received in Washington by Cereal Chemistry and the periodic forwarding of this money to the

Secretary-Treasurer.

The Executive Committee has appropriated a sum of money not to exceed \$250.00 to be spent during the coming year at the discretion of the President and the Executive Committee for the publication and distribution to members only of additional information not regularly occurring in Cereal Chemistry.

The Committee on account of economic conditions believes that it is not ad-

visable at this time to push the Insurance Plan for members.

The Executive Committee recommends that no change be made in the Constitution at present in regard to the election of officers by mail instead of at annual

The Committee recommends the filing of notices of withdrawal for the \$2,000.00 worth of stock in the Benefit Building & Loan Association of Kansas City and for the \$500.00 worth of stock in the Omaha Building and Loan Association of Omaha, and when these funds are received, recommends that they be invested in Government Securities.

The Committee recommends that no reduction be made in the dues of the Association.

Chicago has been selected as the meeting place for the 1933 Convention.

As Chairman of the Executive Committee, I wish to take this opportunity to express my appreciation to the other members for their excellent cooperation throughout the year.

E. B. Working moved that the report of the Executive Committee be accepted. Seconded, carried.

#### Report of the Employment Committee

#### C. B. Morison, Chairman

Letters, correspondence from May 18, 1931 to May 23, 1932		130
Positions filled		
Employers requesting assistance		10
Names registered this year from May 18, 1931 to May 23, 1932	25	
Previous names registered	165	
	-	
T - 1		400

C. B. Morison moved that the report of the Employment Committee be accepted. Seconded, carried.

#### Report of the Committee on Milling Chemistry, Rosenwald Museum of Science and Industry

#### F. L. Dunlap, Chairman

The past year has been for your Committee on Milling Chemistry a period of "watchful waiting." Inasmuch as no request has been made of your Committee for cooperative work in this field of Milling Chemistry, there is no special report that can be rendered.

C. B. Morison read this report in the absence of F. L. Dunlap who is recovering from a serious accident. C. B. Morison moved that the report be accepted. Sec-

C. G. Harrel made a short informal report of the work of the Publicity Committee during the year and moved that the report be accepted. Seconded, carried.

#### Report of History Committee

#### R. Wallace Mitchell, Chairman

The last report of this Committee was accompanied by the manuscript of the "History" up to the year 1924. This manuscript was submitted to the Executive Committee for criticism and suggestion. The idea being that the plan of presenting the material should be approved before completing the record for the ensuing years.

The comments of the Executive Committee members have been constructive and have resulted in making the record more satisfactory.

We submit the finished manuscript complete for the period 1915 to 1930 for

your approval.

This Committee has not been satisfied with its own efforts in writing the record for the last several years. We feel that the data might very possibly be assembled and presented more effectively by a Committee composed of members who have served in office during recent years. This suggestion is made in the interest of a history that will carry into the record the spirit and atmosphere of the period.

For these reasons we wish to recommend to the officers that they appoint a new committee to bring this history up to date and provide for the continuance of the work.

J. A. Dunn moved that the report of the History Committee be accepted. Seconded, carried.

C. H. Bailey moved that the Secretary be instructed to write a letter of thanks to R. W. Mitchell expressing the appreciation of the members of the Association for the work that he and his committee have done in compiling the history of the Association from 1915 to 1930. Seconded, carried.

#### Report of the Membership Committee

#### L. E. Leatherock, Chairman

The Membership Committee carried on an active campaign by mail and personal solicitation. Most of the members took an active part. We believe that a large percentage of the eligible prospects were solicited.

The Corporation Membership privilege was brought to the attention of a large number of eligible firms, but the results were not up to expectations, partly due to the present conditions.

The Pioneer Section revised their by-laws to exclude from their check sample list firms and individuals not members of the A. A. C. C. This action proved to be the biggest help the committee had in securing new members.

#### Report of the Committee on Resolutions

#### Paul Logue, Chairman

Whereas, the American Association of Cereal Chemists has been privileged to hold another successful convention, and

Whereas, the success of this convention has been due largely to the faithful

service rendered by the officers and committees of this association,

Therefore, be it resolved, that the thanks of this association be extended to our President, R. K. Durham; our Vice-President, L. D. Whiting; our Secretary-Treasurer, M. D. Mize; the Programme Committee, Ralph M. Bohn, Chairman; the Local Arrangements Committee, Raymond T. Bohn, Chairman; the Methods Committee, C. E. Mangels, Chairman; the Committee on Standardization of Laboratory Baking, D. A. Coleman, Chairman; the Committee on Methods of Testing Cake and Biscuit Flours, Mary M. Brooke, Chairman, and all other committees

or members who have contributed toward the success of this convention.

Be it further resolved, that we express our thanks to Dr. B. J. Bush, for his

invocation at the opening of the convention.

Be it resolved, that we express our thanks to J. Lee Barrett of the Detroit

Convention and Tourists Bureau for the cordial welcome to Detroit.

Be it resolved, that we express our appreciation of the greeting extended us by the Association of Operative Millers through their President, H. H. Trapp, and their Secretary C. W. Partridge.

Be it resolved, that the thanks of this Association be extended to Bausch and Lomb for the use of the Baloptican and the educational reel it so generously furnished.

Be it resolved, that we express our thanks to C. G. Harrel for the permanent record of faces and features he has so industriously made and graciously shown of the convention activities, and the efficient manner in which he has handled the projection requirements of the speakers.

Be it also resolved, that we express our appreciation of the cooperation of the local section of the American Chemical Society.

Be it resolved, that the thanks of this convention be extended to the:

Central Scientific Company Laboratory Construction Company Provident Chemical Works Standard Brands, Inc. Victor Chemical Works Wallace & Tiernan Co., Inc. Wesson Oil and Snowdrift Sales Co.

for furnishing the golf trophies.

Be it resolved, that the appreciation of the Association be expressed to the David Stott Flour Mills, the Commercial Milling Company and F. W. Stock & Sons for the delightful entertainment provided the ladies and to the Ladies Entertainment Committee, Mrs. R. T. Bohn, Chairman, for its effective management. Be it resolved, that we express our appreciation to the Statler Hotel, J. H.

Pickler, Manager, for their kind hospitality in taking care of the convention and of

us individually.

Whereas, misfortune has befallen our fellow member, Dr. F. L. Dunlap,

Be it resolved, that this Association extend to him its deep sympathy and

Be it resolved, that our sympathies and wishes for speedy recovery be conveyed to our fellow member, C. B. Kress, who has been prevented from meeting with us because of a serious operation.

Be it further resolved, that out of respect for our late fellow member, Robert Dewar, who during the year has passed from our midst, the Association in convention

now assembled stand for a moment in silence.

Paul Logue moved that the resolutions be adopted, seconded.

C. E. Mangels amended the motion to the effect that all reference to the Eighteenth Amendment be eliminated. Seconded, carried.

The original motion was then carried.

#### Report of the Auditing Committee

V. E. Fisher, Chairman

The Auditing Committee has examined the records of our Secretary-Treasurer for the fiscal year, January 1st, 1931 to December 31st, 1931 and find same to be correct.

We recommend that the Executive Committee consider the advisability of an annual examination of the physical properties of the Association for the convenience

of future auditing committees.

V. E. Fisher moved that the report of the Auditing Committee be accepted. Seconded, carried.

#### Report of the Nominating Committee

M. J. Blish, Chairman

Election of officers:

President-L. D. Whiting. Vice-President—R. C. Sherwood. Secretary-Treasurer—M. D. Mize. Editor-in-Chief Cereal Chemistry—D. A. Coleman. Managing Editor Cereal Chemistry—C. C. Fifield.

Washington Platt moved that a committee be appointed to propose tentative definitions of technical terms used by cereal chemists, seconded.

J. A. Dunn amended the motion so as to read that this committee make a report of such definitions so that it can be printed in an issue of the News Letter three months prior to the 1933 Convention. Seconded, carried.

The motion was then carried as amended.

A. W. Meyer moved that a committee be appointed to study methods of testing and baking of rye flour. Seconded, carried.

Report of the Committee on Methods of Analysis, C. E. Mangels, Chairman. Sub-report by C. F. Davis, "Magnesium-Acetate-Alcohol Method of Determining Ash" read by H. W. Putnam in the absence of C. F. Davis.

Sub-report by A. E. Treloar, "Statistical Analysis of Cereal-Chemical Analytical

Data.

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C. E. Mangels moved that the recommendations given in the Report of the Methods of Analysis Committee, page 429, this issue of Cereal Chemistry, be adopted. Seconded and carried.

M. A. Gray moved that a special committee be appointed to consult and cooperate with the Millers' National Federation and others in establishing a moisture basis for reporting chemical constituents of wheat, flour, and feeds. Seconded,

D. A. Coleman moved that recommendations of the Committee of Standardization of Laboratory Baking given on page 405 of this issue of Cereal Chemistry, be adopted. Seconded, and carried. Meeting adjourned at 5:30 p.m.

## Thursday, May 26

Meeting was called to order by President Durham at 9:00 a.m. and placed in charge of the Chairman for the morning, C. B. Morison.

Paper—"Measuring and Recording Some Characteristics of Test Sponge Cakes" by Washington Platt and P. D. Kratz. Washington Platt read this paper and gave some demonstrations of the measurement of toughness in cake.

Paper—"Some Factors Which Affect the Gas Production in a Bread Dough" by W. L. Heald.

Paper—"Sugar, Diastatic Activity, and Gassing Power of Wheat Flour" by M. J. Blish and R. M. Sandstedt, read by M. J. Blish.

Paper—"Some Aspects of the Bromate Baking Tests" by W. F. Geddes and

R. K. Larmour, read by W. F. Geddes.

Paper—"Photochemical Action, a Cause of Rancidity in Foods" by M. R.

Coe and J. A. LeClerc, read by Bert D. Ingels.

L. D. Whiting moved that the secretary-treasurer be given one hundred dollars as a partial appreciation of the work done during the past year.

Meeting delaymed 12 00 needs.

Meeting adjourned 12.00 noon. Meeting called to order by President Whiting at 2:00 p.m.

C. G. Harrel exhibited some motion pictures of the action doughs and of bread during baking.

The meeting was then placed in charge of R. M. Bohn, Chairman for the after-

Paper—"Biological Method for the Determination of Different Sugars and Starch Degradation Products" by A. S. Schultz and G. W. Kirby, read by G. W.

Paper—"Investigation of a Mercury Vapor Lamp for Accelerating the Bleaching Action of Dibenzoyl Peroxide" by C. G. Ferrari and Alice B. Croze, read by C. H.

Paper-"Studies of Doughs with the Brabender Farinograph" by Oscar Skov-

holt and C. H. Bailey, read by Oscar Skovholt. Paper—"What American Export Wheat and Flour Types are Best Suited for Mixing with European Soft Wheat Types" by C. W. Brabender, read by Arthur Hartkopf.

Paper—"A Study of Proteins of Wheat Bran" by G. L. and W. K. Teller. President Whiting appointed the following standing committees for the year:

#### **Executive Committee**

R. C. Sherwood, Chairman Bert D. Ingels R. K. Durham C. G. Harrel L. D. Whiting

#### Membership Committee

W. A. Richards, Chairman W. L. Heald R. B. Potts T. E. Carl L. R. Olsen W. O. Whitcomb C. O. Oppen L. E. Erlenbaugh G. R. Stadler W. G. Epstein

#### Committee on Methods of Analysis

C. E. Mangels, Chairman A. W. Meyer J. T. Flohil F. A. Collatz A. E. Treloar C. F. Davis G. L. Alexander

Committee on Standardization of	Laboratory	Baking
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W. F. Geddes, Chairman	M. J. Blish
R. K. Larmour	C. H. Bailey
C. O. Swanson	R. T. Bohn
C. N. Frey	G. F. Garnatz
D. A. Coleman	

#### Committee on Methods of Testing Cake Flours

Mary M. Brooke, Chairman	G. L. Alexander
L. H. Bailey	R. A. Barackman
E. E. Smith	Pearl Brown
J. A. Dunn	R. T. Bohn
J. W. Montzheimer	H. G. L. Walter
R. A. Pouchain	W. E. Stokes

#### Committee on Testing Rye Flours

L. H. Bailey, Chai G. Moen	rman	W. G. Eps T. W. San	
		T. W. San	

#### Committee on Pie Flours

A. A. Schaal, Chairman	P. E. Minton
M. B. Graff	C. B. Kress
G. N. Bruce	W. K. Marshall

#### Committee on Definitions of Technical Terms

Washington Platt, Chairman	M. A. Gray
C. B. Morison	C. O. Oppen
Ouick Landis	I. A. LeClerc

## \* Committee on Employment

nttee on Emplo	yment	
C. B. Morison	Chairman	M. D. Mize

## Committee on Publicity

interest on a montorey	
C. G. Harrel, Chairman	Washington Platt
Rert D. Ingels	H. W. Putnam

#### Committee on Osborne Medal Award

Paul Logue, Chairman	H. E. Weaver
C. L. Alsberg	C. O. Swanson
M. A. Gray	

## Committee on Milling Chemistry at Julius Rosenwald Museum of Science and Industry

F. L. Dunlap, Chairman H. E. Weaver	C.	В.	Morison
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## Convention Peptizer

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#### Committee on Definition of Moisture Basis for Laboratory Reports

R. K. Durham, Chairman	F. A. Collatz
M. A. Gray	R. J. Clark
I A LoClore	

#### Program Committee for 1933 Convention

C. B. Morison, Chairman	C. H. Bailey
W. F. Geddes	C. O. Swanson
C. S. Miner	R. M. Bohn

## Local Arrangements Committee for 1933 Convention

#### REGISTRATION OF CONVENTION, DETROIT, MICHIGAN MAY 23-MAY 26, 1932

#### Members

MAY 23—MAY 26, 1932

Members

Geo. L. Alexander, Commercial Milling Co., Detroit, Mich. Lowell Armstrong, Ballard & Ballard Co., Louisville, Ky.

C. H. Bailey, University of Minnesota, St. Paul, Minn. John C. Baker, Wallace-Tierna & Co., Newark, N. J. R. A. Barackman, Victor Chemical Works. Micago Heights, Ill. R. A. Barackman, Victor Chemical Works. Micago Heights, Ill. R. A. Barackman, Victor Chemical Works. Micago Heights, Ill. R. A. Barackman, Victor Chemical Works. Micago Heights, Ill. R. A. Barackman, Victor Chemical Works. Mich. H. M. Blinn, 3401 Greenway, Baltimore, Md.

M. J. Blish, Agri. College, Univ. of Nebr., Lincoln, Nebr. Leonard J. Bohn, Trent Institute, O.A.C., Guelph, Ont., Canada. Ralph M. Bohn, Independent Biscuit Mgrs. Co., Chicago, Ill. Raymond T. Bohn, A. & P. Tea Co., Detroit, Mich. D. L. Boyer, Provident Chemical Works, St. Louis, Mo. Mary Minton Brooke, Purity Bakeries Corp., 844 Rush St., Chicago, Ill. Pearl Brown, Perfection Biscuit Co., Fort Wayne, Ind. H. J. Brownlee, United Mills Co., Inc., Grafton, Ohio. R. D. Callaway, Laboratory Construction Co., Kansas City, Mo. Theodore E. Carl, Standard Brands, Inc., New York City. Howard A. Clark, Standard Brands, Inc., New York City. Howard A. Clark, Standard Brands, Inc., New York City. D. A. Coleman, U. S. Dept. of Agri., Washington, D. C. William A. Crowe, Central Scientific Co., Chicago, Ill. A. E. Cutris, Midland Flour Milling Co., Kansas City, Mo. Rudy S. Edel, Bay State Milling Co., Kansas City, Mo. Rudy S. Edel, Bay State Milling Co., Chassa, Milling Co., Chicago, Ill. C. C. Fifield, U. S. Dept. of Agri., Washington, D. C. William, A. Crowe, Central Scientific Co., Chicago, Ill. C. C. Fifield, U. S. Dept. of Agri., Washington, D. C. William, Co., Minneapolis, Minn. L. W. Francis, G. R. R. Smith Milling Co., Minneapolis, Minn. L. W. Francis, G. R. R. Smith Milling Co., Minneapolis, Minn. L. W. Francis, G. R. R. Smith Milling Co., Minneapolis, Minn. L. W. Francis, C. R. Smith Milling Co., Port Colborne, Onto. Charles, Milling Co., Mi

R. Augustin Pouchain, Tasty Baking Co., Philadelphia, Pa. H. W. Putnam, Hays City Flour Mills, Hays, Kansas. O. H. Raschke, Victor Chemical Works, Chicago, Ill. W. A. Richards, International Milling Co., Buffalo, N. Y. R. M. Sandstedt, University of Nebr., Lincoln, Nebr. A. A. Schaal, Lever Bros., Co., Cambridge, Mass. R. C. Sherwood, General Mills, Inc., St. Paul, Minn. Oscar Skovholt, University Farm, St. Paul, Minn. Edward E. Smith, F. W. Stock & Sons, Hillsdale, Mich. W. E. Stokes, Royal Baking Powder Co., Brooklyn, N. Y. W. Kedzie Teller, The Columbus Lab., Chicago, Ill. Adrian Thomas, Huron Milling Co., Harbor Beach, Mich. E. F. Tibbling, Washburn Crosby Co., Kansas City, Mo. Alan E. Treloar, Uni. of Minn., St. Paul, Minn. Edgar L. Ulrey, Acme-Evans Co., Indianapolis, Ind. E. Rex Uphouse, Hall Milling Co., St. Louis, Mo. H. F. Vaupel, Washburn Crosby Co., Inc., Chicago, Ill. H. G. L. Walter, Igleheart Bros., Inc., Evansville, Ind. A. G. O. Whiteside, Cereal Div., Experimental Farm, Ottawa, Canada. L. D. Whiting, Ballard & Ballard Co., Louisville, Ky. A. K. Whittaker, David Stott Flour Mills, Detroit, Mich. H. Kenneth Wilder, Kellogg Co., Battle Creek, Mich. O. B. Winter, Mich. State College, E. Lansing, Mich. Earl B. Working, Kansas State College, Manhattan, Kansas. W. B. Young, State Protein Lab., Minneapolis, Minn.

#### Visitors and Guests

T. H. Barnard, Clinton Corn Syrup Refining Co., Clinton, Iowa. Lydia Cooley, Calumet Baking Powder Co., Chicago, Ill. Wm. L. Fawcett, The Hobart Mfg. Co., Troy, Ohio. Arthur Hartkopf, Brabender Corp., Minneapolis, Minn. J. P. Henderson, C. J. Tagliabue Mfg. Co., Brooklyn, N. Y. Geo, W. Kirby, Standard Brands, Inc., New York, City. Quick Landis, Fleischmann Labs., New York, N. Y. Elizabeth McKim, Provident Chemical Works, St. Louis, Mo. Gordon Mainland, Kistler Automatic Sampler Co., Longmont, Colo. Edwin Moniot, Thomson Machine Co., Belleville, N. J. Fred Weberpals, H. C. Bohack Co., New York City. John R. White, Lever Bros., Co., Cambridge, Mass.

John R. White, Lever Bros., Co., Cambridge, Mass Mrs. Lowell Armstrong, Louisville, Ky. Mrs. Ralph M. Bohn, Chicago, Ill. Miss Georgia Bohn, Chicago, Ill. Miss Georgia Bohn, Chicago, Ill. Mrs. R. T. Bohn, Detroit, Mich. Mrs. Ethel Brownlee, Cedar Rapids, Ia. Mrs. W. L. Fawcett, Troy, Ohio. Mrs. E. N. Frank, Minneapolis, Minn. Mrs. Geo. Garnatz, Cincinnati, Ohio. Mrs. W. F. Geddes, Winnipeg, Man., Canada. Mrs. M. A. Gray, Minneapolis, Minn. Mrs. N. L. Gregory, Port Colborne, Ont., Canada. Mrs. C. G. Harrel, Minneapolis, Minn. Mrs. L. E. Jackson, Chicago, Ill. Mrs. S. J. Lawellin, New Ulm, Minn. Mrs. L. E. Leatherock, Wichita, Kans. Mrs. L. E. Leatherock, Wichita, Kans. Mrs. J. M. Lugenbeel & Daughter, St. Louis, Mo.

Mrs. R. M. McKinstrie, Troy, Ohio,
Mrs. F. D. Machon, Detroit, Mich.
Mrs. A. G. Olsen, Battle Creek, Mich.
Mrs. E. M. Paget, Chicago, Ill.
Mrs. E. C. Paulsel, Minneapolis, Minn.
Mrs. R. B. Potts, Wichita, Kans.
Mrs. R. A. Pouchain, Philadelphia, Pa.
Mrs. H. W. Putnam, Hays, Kans.
Mrs. O. H. Raschke, Chicago, Ill.
Mrs. W. A. Richards, Buffalo, N. Y.
Mrs. R. C. Sherwood, St. Paul, Minn.
Mrs. Ed. E. Smith, Hillsdale, Mich.
Mrs. W. K. Teller, Chicago, Ill.
Mrs. Adrian Thomas, Harbor Beach, Mich.
Mrs. A. E. Treloar, St. Paul, Minn.
Mrs. O. B. Winter, E. Lansing, Mich.

#### **BULLETIN REVIEWS**

Fifth Annual Report (1931), Dominion Grain Research Laboratory, Department of Trade and Commerce, Board of Grain Commissioners for Canada, Winnipeg, Manitoba, 1932. 43 pp.

Investigational and service activities are reported for the year 1931. Under investigational activities data are shown with respect to the protein content of the contract grades of hard red spring wheat grown in western Canada during the year 1931; for the maximum, minimum, and average protein content of the contract grades of wheat grown in the three prairie provinces for the five years 1927–31; for the quantity and quality of oil produced from different types and varieties of flax-seed; and for the results of a study to determine the suitability of electrical moisture tests for the determination of moisture in wheat for grain inspection purposes.

With respect to service activities, the results of extensive studies are presented for the purposes of grain inspection and grain standardization. Data are shown recording findings on the milling and baking properties of the 1931 crop of Canadian spring wheat, the effect of drying, as well as the effect of spring thrashing upon the milling and baking properties of wheat.

Studies on durum wheat, particularly with reference to black tip fungus, are also

presented.

D. A. COLEMAN.

Second Annual Report, Wheat Research Institute, New Zealand Department of Scientific and Industrial Research, Christchurch. Bulletin No. 35, December, 1931. 50 pp.

This report covers a large number of subjects, the scope of which is too extensive to detail here. Broadly speaking, the data presented record the results of plant breeding experiments; milling, baking, and chemical studies; as well as the results of economic studies with respect to wheat production in New Zealand. For the cereal chemist the section on milling, baking, and chemical investigation is very instructive. Summaries of results are shown covering the following: Protein survey, milling and baking quality of varieties, testing wheats for millers and bakers, milling and baking quality of wheat cut at various stages of maturity, baking qualities of header harvested wheats, moisture testing studies, effect of manures on the milling and baking quality of wheats, studies on the aging of flour, effect of repeated wetting and drying of wheat on its bushel weight and milling quality, the effect of heating special fractions of flour on baking quality, studies on the diastatic activity of flours, and the analyses of stock feeds.

D. A. COLEMAN.

The Use of Oils, Fats, and Emulsions of Oils and Fats in Bread Making, by E. A. Fisher and C. R. Jones. Reprint from National Association Review, April 1 and 8, 1932. Melbourne House, Aldwych, London. 8 pp.

The report describes extensive experiments carried out on the use of oils and fats and emulsions of oils and fats in breadmaking. The oils and fats studied were coconut, olive, palm kernel, peanut, flour, castor, medicinal paraffin, lard, and shea butter. They were used in varying proportions both in the "raw" state and in the form of emulsions. Emulsifying agents were employed to produce stable emulsions. Coconut oil and medicinal paraffin produced marked deterioration in bread quality, while olive, peanut and palm kernel oil produced very slight improvement in bread quality. The emulsifying agents alone produced slight but definite deterioration in bread quality. When emulsions instead of "raw" unemulsified oils were used deterioration caused by paraffin and coconut oil became more marked and the slight improvement noted with the use of "raw" olive and peanut oils was lost. Shea butter, castor oil, and particularly lard and flour oil produced striking improvements both when added raw or in the form of emulsions; the improvements being more striking in the instance of the raw oil or fat.

Improvement in bread quality was noticeably less with short patent flours than with straights or low grades.

Only slight effects were observed on dough characteristics. Similarly, there was no effect on yeast fermentation.

On the whole, lard was outstandingly the best of the oils and fats examined. Best results were obtained with 2% of raw lard or 3% of emulsified lard (expressed as percentage of flour).

The findings are contrary to those of earlier workers who report superior bread from the use of emulsified oils.

D. A. COLEMAN.